NOVA

Getting started

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Introduction

Nova is designed to control all the Autolab potentiostat/galvanostat instruments with a USB connection. It is the successor of the GPES/FRA software and integrates two decades of user experience and the latest .NET software technology.

Nova brings more power and more flexibility to the Autolab instrument, without any hardware upgrade.

Nova is designed to answer the demands of both experienced electrochemists and newcomers alike. Setting up an experiment, measuring data and performing data analysis to produce publication ready graphs can be done in a few mouse clicks.

Nova is different from other electrochemical software packages. As all electrochemical experiments are different and unique, Nova provides an innovative and dynamic working environment, capable of adapting itself to fit your experimental requirements.

The design of Nova is based on the latest object-oriented software architecture. Nova is designed to give the user total control of the experimental procedure and a complete flexibility in the setup of the experiment.

This getting started manual provides installation instructions for the Nova software and the Autolab hardware. It also includes a quick walkthrough tutorial and a description of the Autolab procedures. Five chapters are included in this document:

- Chapter 1 provides installation instructions for Nova and the Autolab
- Chapter 2 describes a quick cyclic voltammetry measurement
- Chapter 3 describes the Autolab standard procedures
- Chapter 4 provides information about the Autolab hardware
- Chapter 5 provides information regarding Warranty and Conformity

Warning

Please read the Warranty and Conformity carefully before operating the Autolab equipment.

The philosophy of Nova

Nova differs from most software packages for electrochemistry.

The classic approach used in existing electrochemical applications is to code a number of so-called *Use cases* or *Electrochemical methods* in the software. The advantage of this approach is that it provides a specific solution for well-defined experimental conditions. The disadvantage is that it is not possible to deviate from the methods provided in the software. Moreover, it is not possible to integrate all the possible electrochemical methods, since new experimental protocols are developed on a daily basis. This means that this type of software will require periodical updates and will necessitate significant maintenance efforts.

Figure 1 shows a typical overview of a classic, *method-based* application for electrochemistry.

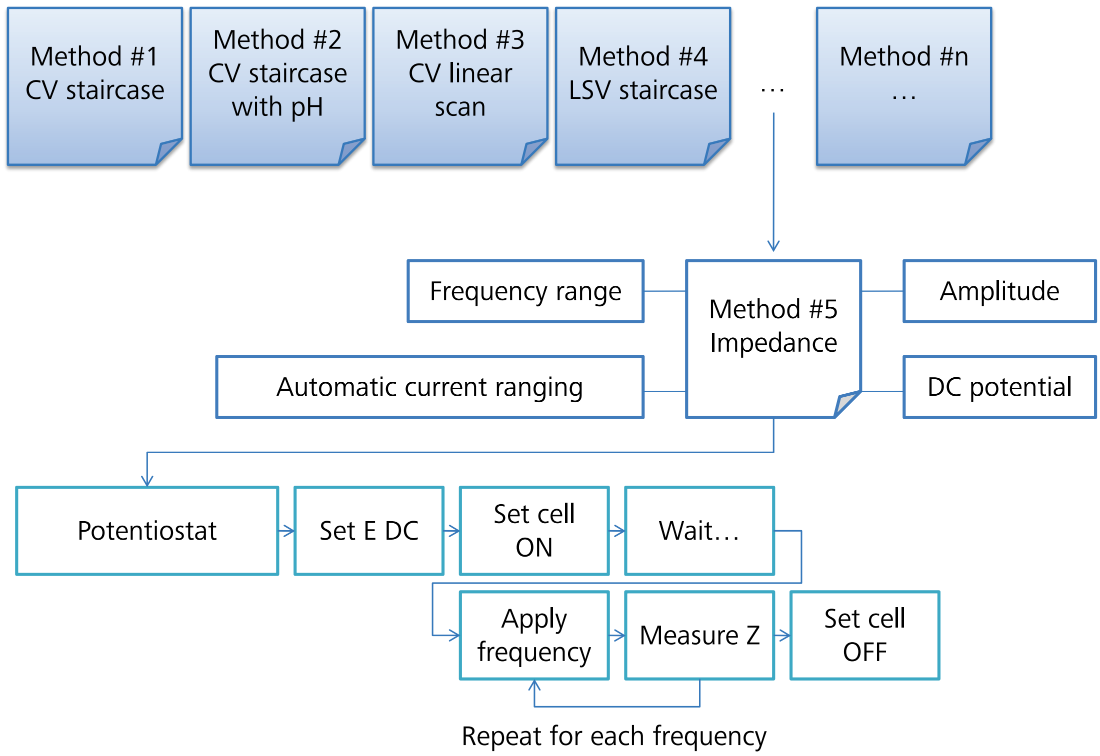


Figure 1 – Schematic overview of a method-based software

In a method-based application, the user chooses one of the *n* available methods and defines the available parameters for the method. When the measurement starts, the whole method is uploaded to the instrument where it is decomposed into individual, low-level instructions. These are then executed sequentially until the measurement is finished.

If the method required by the user is not available, the user will have to wait until the method is implemented in a future release.

Nova has been designed with a completely different philosophy. Rather than implementing well defined methods in the software, Nova provides the users with a number of basic **Objects** corresponding to the low-level functions of the electrochemical instrument. These objects can be used as **building blocks** and can be combined with one another according to the requirements of the user in order to create a complete experimental method. In essence, the scientist uses Nova as a programming language for electrochemistry, building simple or complex procedures out of individual commands. The instructions can be combined in any way the user sees fit. Rather than providing specific electrochemical methods to the user, Nova uses a **generic approach**, in which, in principle, any method or any task can be constructed using the available commands.

Figure 2 shows the Nova strategy, schematically.

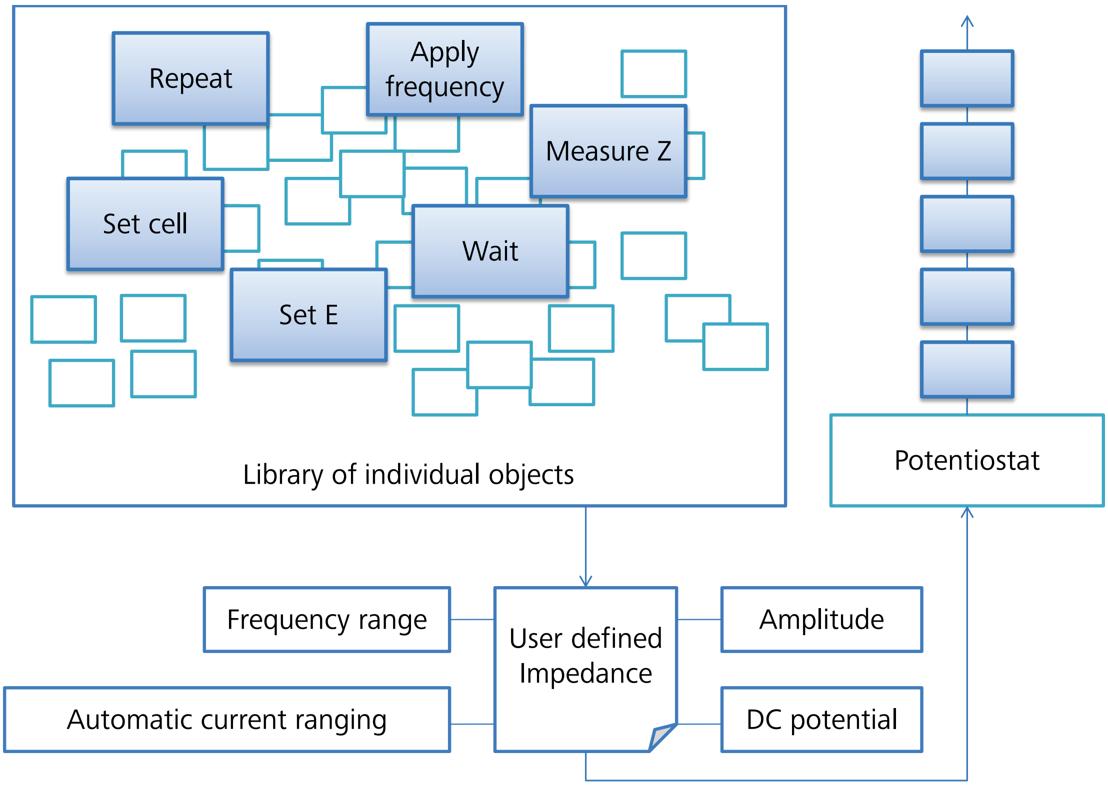


Figure 2 – Schematic overview of the object-based design of Nova

The Nova approach allows the user to program an electrochemical method in the same *language* used by the instrument.

This new object-based design philosophy has led to the current version of Nova. As any task can be solved generically, the software is slightly less intuitive than a method-based application. Depending on the complexity of the experiments, the learning curve can be more or less long. For this reason, we advise you to carefully study this Getting started manual as well as the User manual.

Because of the large number of possibilities provided by this application, it is not possible to include the information required to solve each individual use case.

A number of typical situations are explained using stand-alone tutorials (refer to the Help menu – Tutorials). These tutorials provide practical examples.

In case of missing information, do not hesitate to contact Metrohm Autolab at the dedicated nova@metrohm-autolab.com email address.

Setting up Nova

1 – Nova installation

This chapter describes the steps required for the installation of NOVA and the Autolab instrument.

1.1 – Requirements

Nova requires Windows XP, Windows Vista, Windows 7 or Windows 8 as operating systems in order to run properly. Minimum RAM requirement is 1 GB and the recommended amount is 2 GB. Only the instruments[1] with a USB interface (internal or USB interface box) are supported.

1.2 – Software installation

Warning

Leave the Autolab switched off during the installation of the software.

Insert the Nova CD-ROM in the optical drive of your computer. Open the Windows explorer and browse the contents of the disk. Locate the **Setup.exe** program and double click to install Nova on your hard drive.

Note

Installation of the .NET 4.0 framework is required in order to install Nova. If the .NET framework is already installed on your computer, the install wizard will directly install Nova (skip to Section 1.2.2). Otherwise you will be prompted to accept the installation of the .NET 4.0 framework (see .NET framework installation).

1.2.1 – .NET 4.0 framework installation[2]

The Microsoft .NET Framework is a component of the Microsoft Windows operating system. It provides a large body of pre-coded solutions to common program requirements, and manages the execution of programs written specifically for the framework. The .NET Framework is a key Microsoft offering, and is intended to be used by most new applications created for the Windows platform.

[1] The following hardware is **not** supported in NOVA: µAutolab type I and PSTAT10, instruments with ADC124, DAC124 or DAC168 and FRA modules (1st generation FRA). Contact you Autolab distributor for more information.
[2] Please make sure that your copy of Windows has been updated to the latest version.

If the .NET framework 4.0 installation is required, the following window will be displayed (see Figure 1.1). This package is provided by Microsoft and you can read the license agreement by clicking the *View EULA for printing* button.

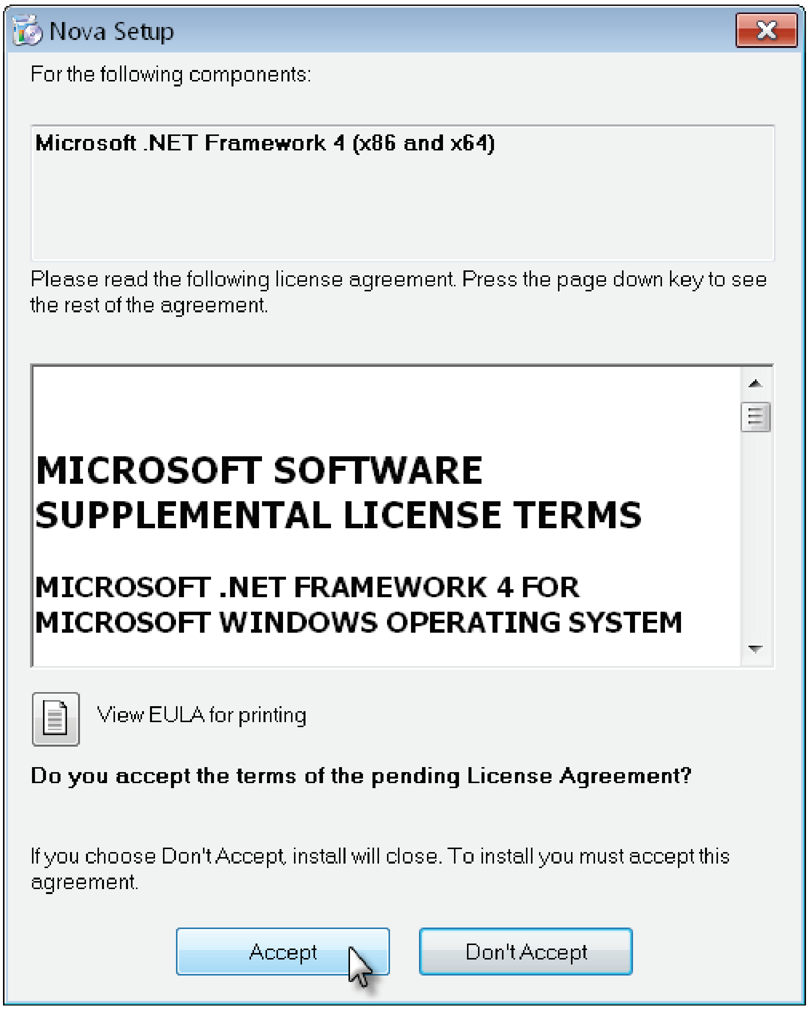


Figure 1.1 – The .NET framework installation wizard

The installation of the .NET framework can take some time. A progress bar is displayed during the installation (see Figure 1.2).

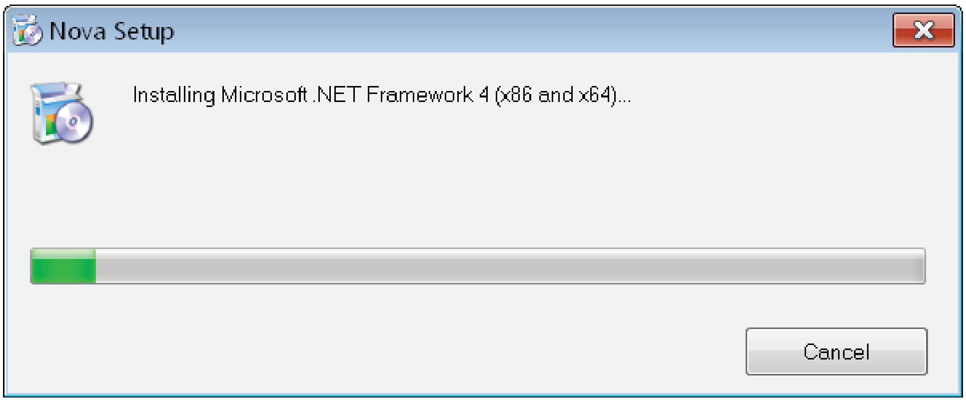


Figure 1.2 – Installing the .NET framework 4.0

When the .NET framework is installed, the installation of Nova will continue.

1.2.2 – Nova installation

If the .NET framework is correctly installed on your computer, the installation wizard starts the setup of Nova (see Figure 1.3).

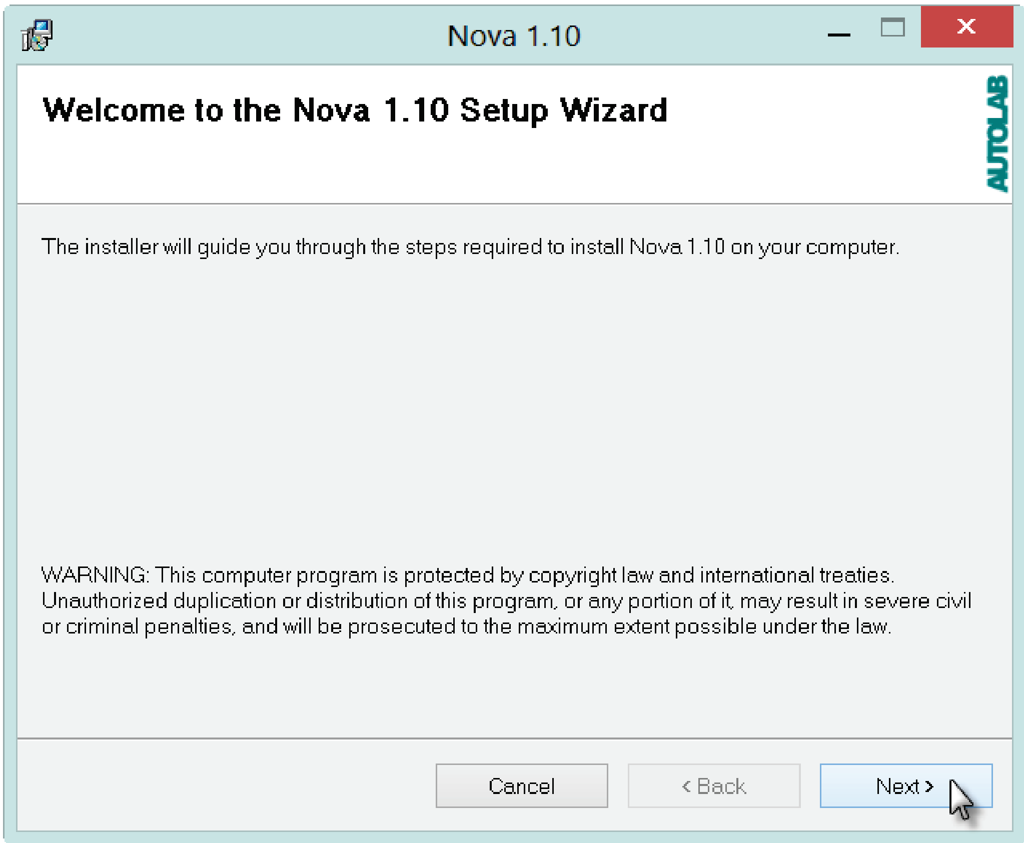
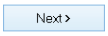


Figure 1.3 – The Nova Setup wizard

Click the 

button to continue the installation. You will be prompted to enter the location of the installation folder or to validate the default setting (see Figure 1.4). Press the 

button to change the installation folder or press the Next button to accept the default.

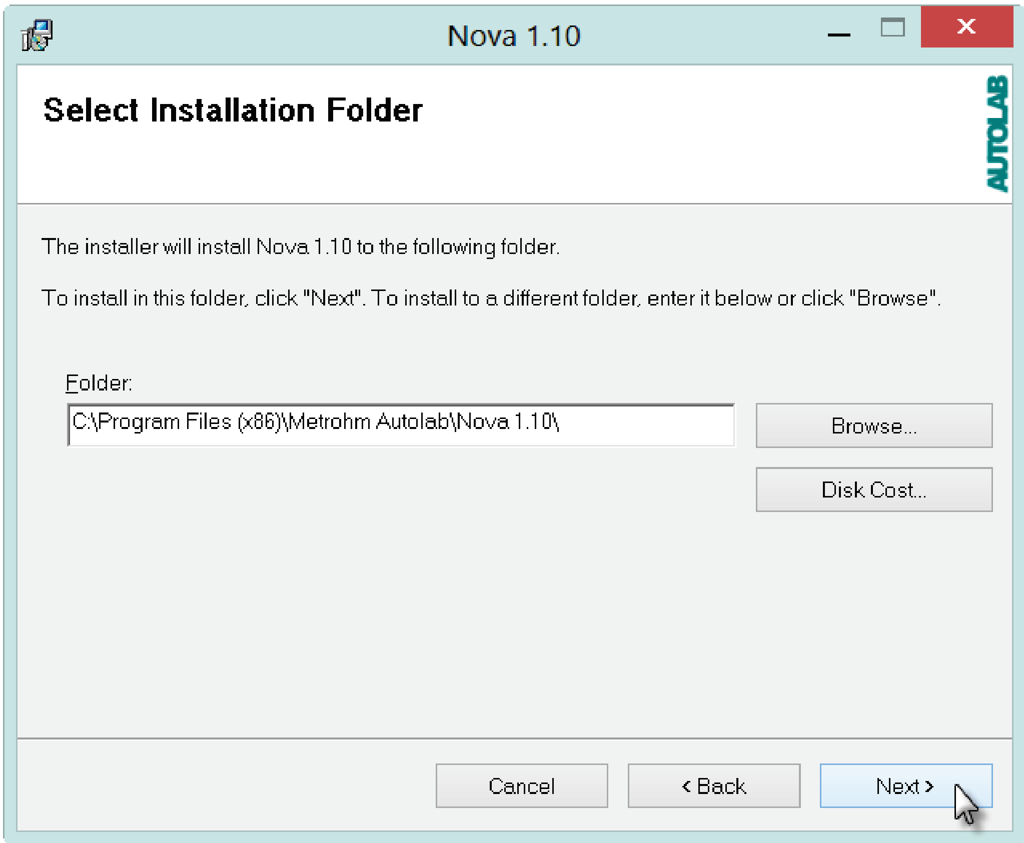


Figure 1.4 – Setting the installation folder

Click the Next > button to confirm the installation of Nova. A progress bar will be displayed during the installation. When the software setup is completed, the Installation Complete window will appear (see Figure 1.5). Click the Close button to finish the installation process.

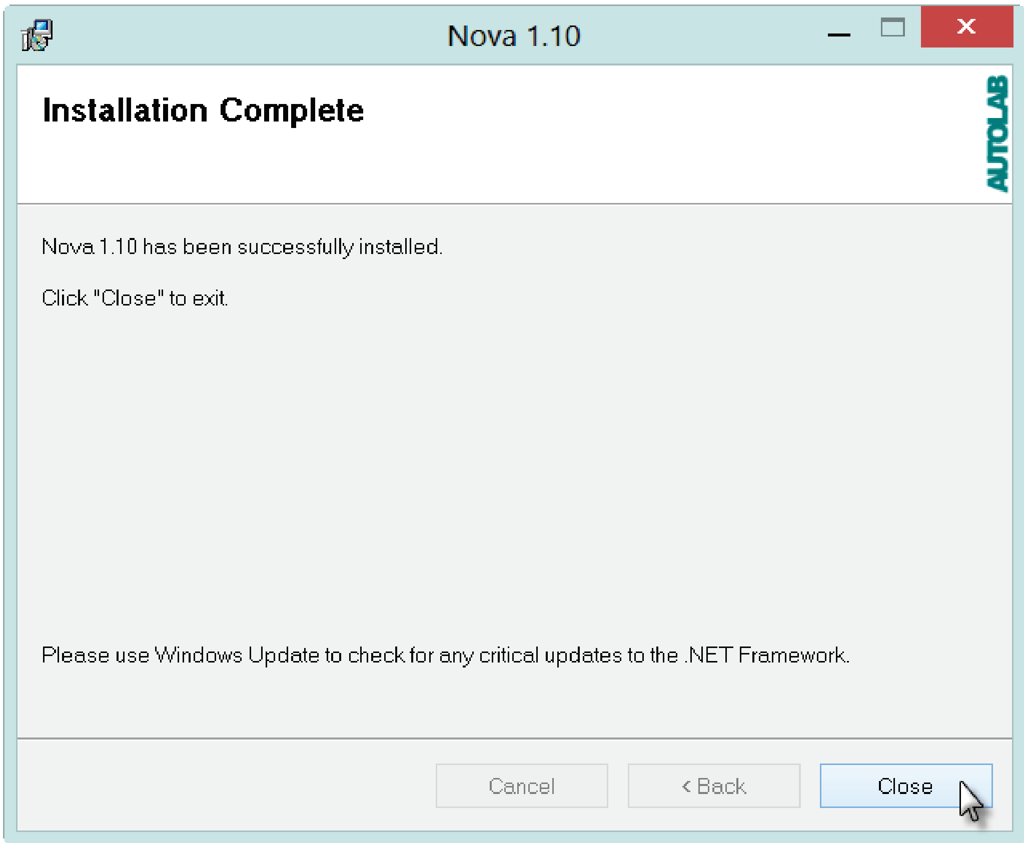


Figure 1.5 – Installation finished

A shortcut to Nova will be added to your desktop or on the Windows 8 menu.

1.2.3 – USB Drivers installation

After Nova has been successfully installed, connect the Autolab instrument to the computer using an available USB port. Switch on the instrument. Windows will attempt to find a suitable driver for the instrument. Since the Autolab is not automatically recognized by Windows, no drivers will be installed at this point.

Start the Autolab Driver manager application by using the shortcut provided in the Start menu (All Programs – Autolab – Tools – Driver manager 1.10) or by using the shortcut tile on the Windows 8 Menu (see Figure 1.6).

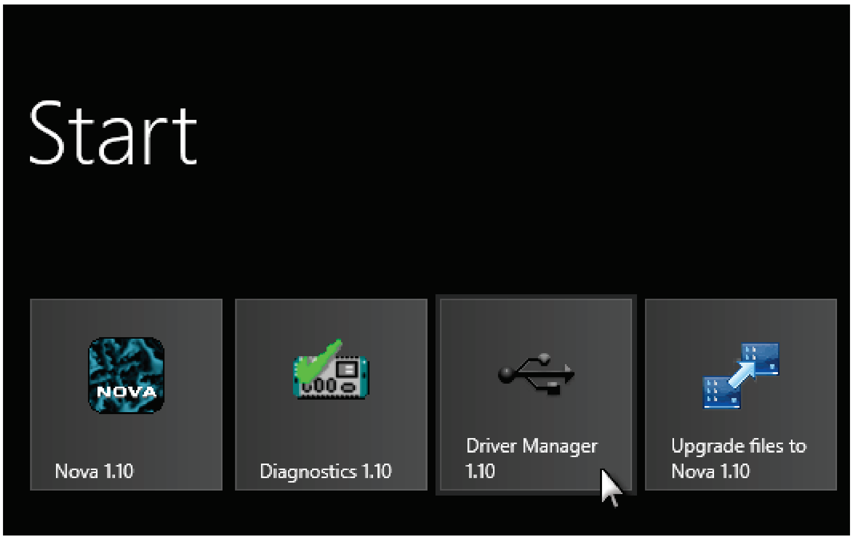


Figure 1.6 – Use the shortcut tile to start the Driver Manager application

This will start the Driver Manager application (see Figure 1.7).

Figure 1.7 – The driver manager application

The Driver Manager can be used at any time to select the driver to use to control the Autolab.

Two drivers are available:

- **NOVA only (recommended setup):** this is the latest driver for the Autolab, allowing up to 16 instruments to be connected to the computer and faster data transfer. This driver is compatible with 64 Bit versions of Windows.
- **GPES compatible:** this is an older driver version. No further developments are planned for this driver. The maximum number of devices connected to the same computer is 8. Data transfer is slower than with the NOVA only driver. This driver is only compatible with 32 Bit versions of Windows.

Warning

The GPES compatible driver is **not** available on a 64 Bit version of Windows.

Warning

The GPES and FRA software only work using the GPES compatible driver.

To install one of the drivers, click either one of the buttons in the Driver manager (see Figure 1.8).

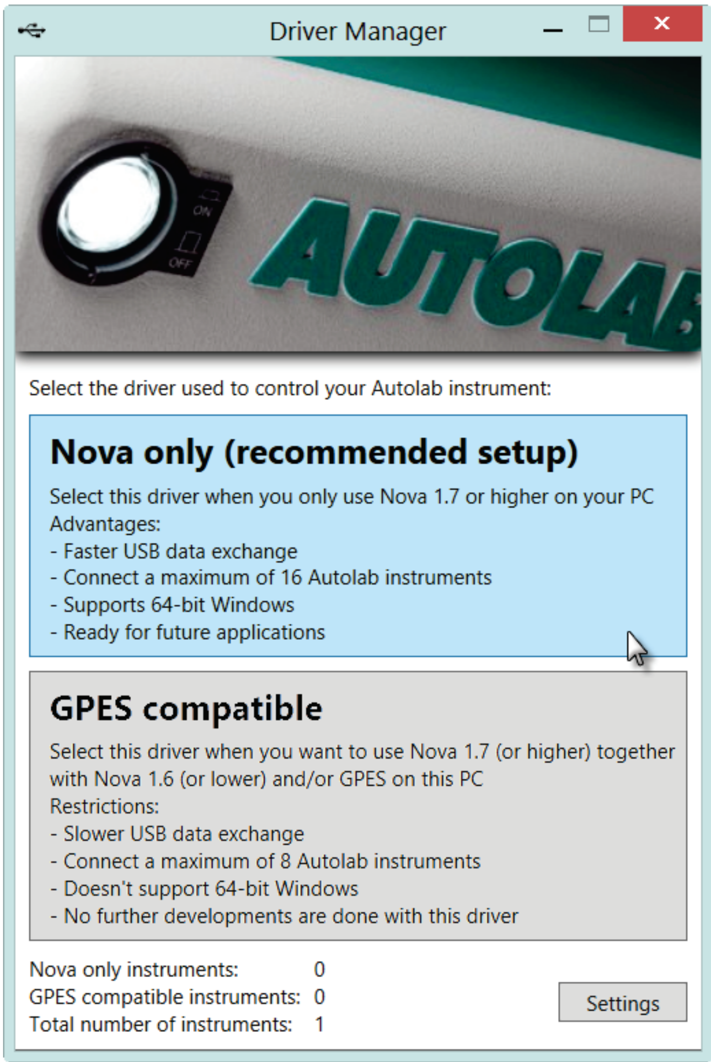


Figure 1.8 – Click one of the two buttons of the Driver Manager to install the driver

Note

The NOVA only driver should be preferably installed.

A message will be displayed, as shown in Figure 1.9.

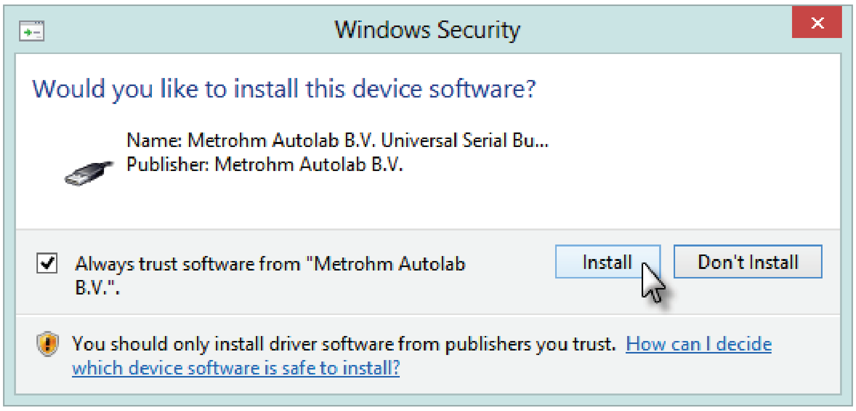


Figure 1.9 – Click the Install button to install the driver software

When the driver installation is completed, a message will be displayed (see Figure 1.10).

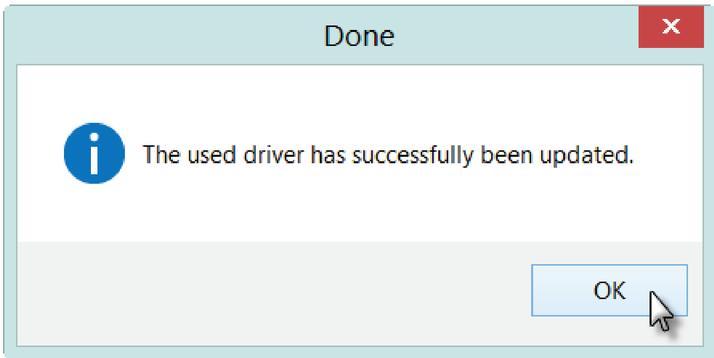


Figure 1.10 – A message is displayed when the installation of the driver is finished

1.2.4 – GPES/FRA and older NOVA versions compatibility[3]

The driver installation description provided in the previous section installs **NOVA only** drivers on the computer. These drivers are **not compatible** with the old Autolab GPES or FRA software and with previous versions of NOVA (NOVA 1.6 and older).

Note

If no connection can be established with the Autolab when using GPES/FRA or older versions of NOVA, check the selected driver using the Autolab Driver Manager.

If necessary, it is possible to use the GPES compatible driver. This driver can be selected at any time using the Autolab **Driver Manager** installed on the computer.

[3] Read this section carefully if you are using GPES/FRA or older versions of NOVA on the same computer.

The Driver Manager is displayed in Figure 1.11. It can be used at any time to select the driver to use to control the Autolab.

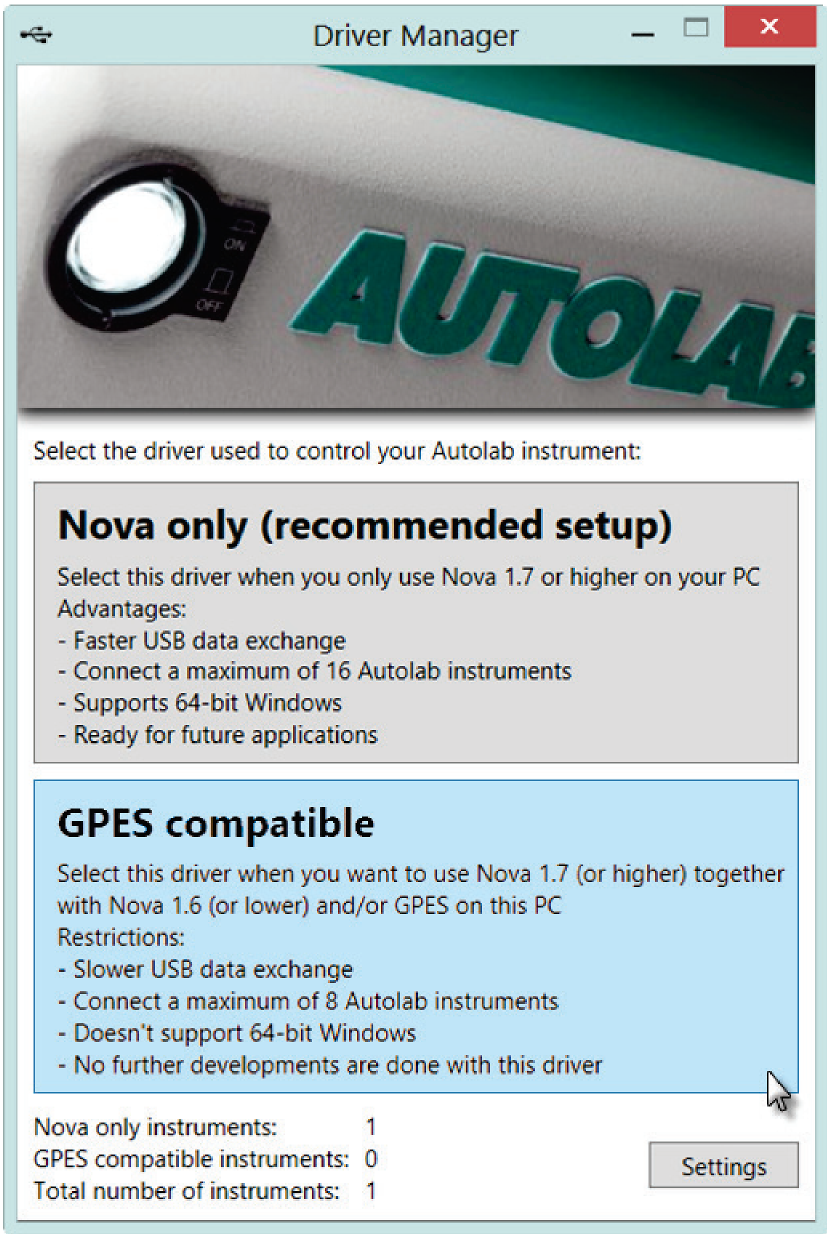


Figure 1.11 – The Autolab Driver Manager can be used to switch drivers

Clicking the GPES compatible button will trigger the installation of the GPES compatible driver for the connected instrument.

Warning

The GPES compatible driver is **not** available on a 64 Bit version of Windows.

A warning will be displayed indicating that the driver cannot be verified (see Figure 1.12).

Figure 1.12 – A warning is provided when the GPES compatible driver is installed

Select the *Install this driver software anyway* option to proceed with the installation. At the end of the installation, a message will be displayed indicating that the driver has been successfully installed (see Figure 1.13).

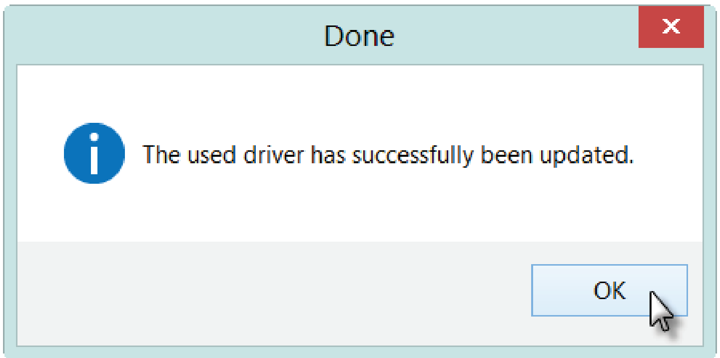


Figure 1.13 – A message is displayed at the end of the driver update process

Note

The status of the drivers used to control the connected devices, displayed at the bottom of the driver manager window, is updated automatically (see Figure 1.14).

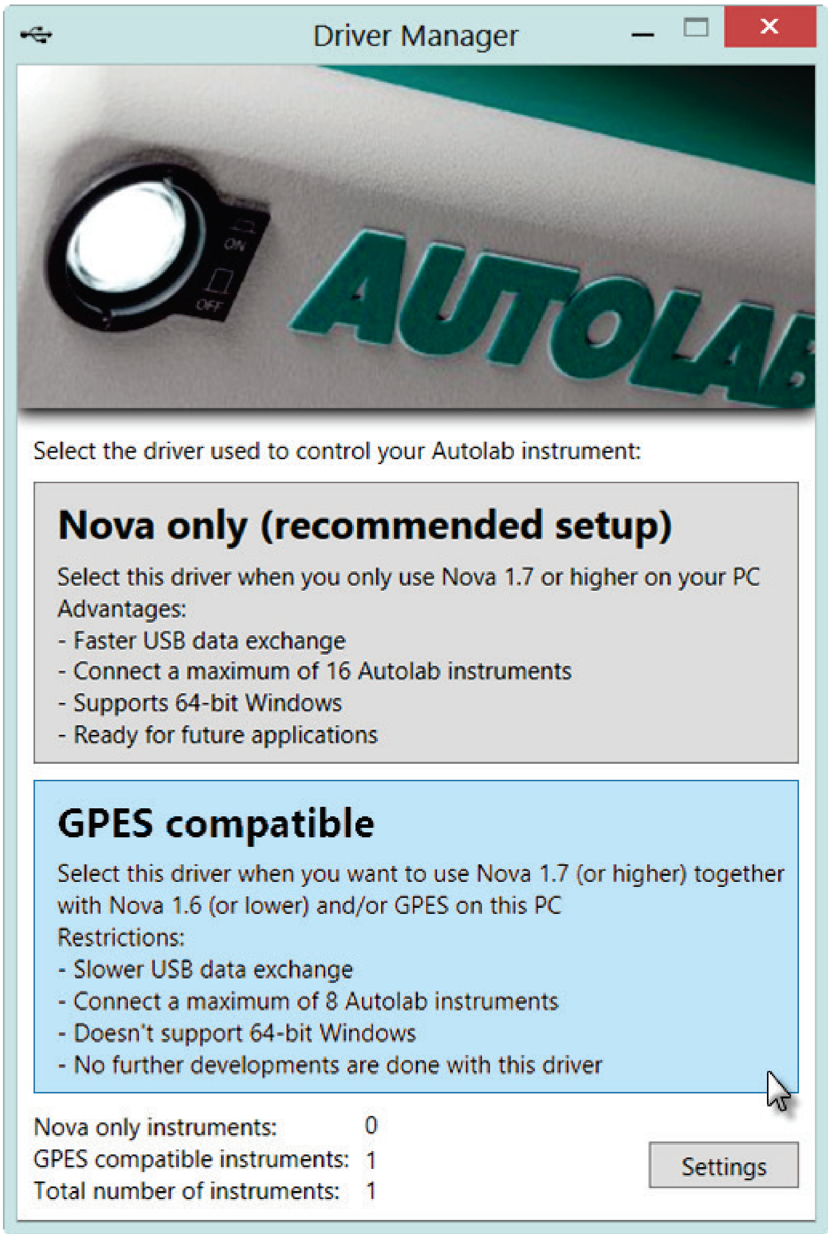


Figure 1.14 – The Driver Manager displays the driver information at the bottom of the window

1.2.5 – Multiple instruments

When the Driver Manager is used on a computer connected to multiple Autolab devices, the drivers will be updated for all the instruments. For example, Figure 1.15 shows that two instruments are connected to the computer. One device is using the NOVA only driver, while the other one is using the GPES compatible driver.

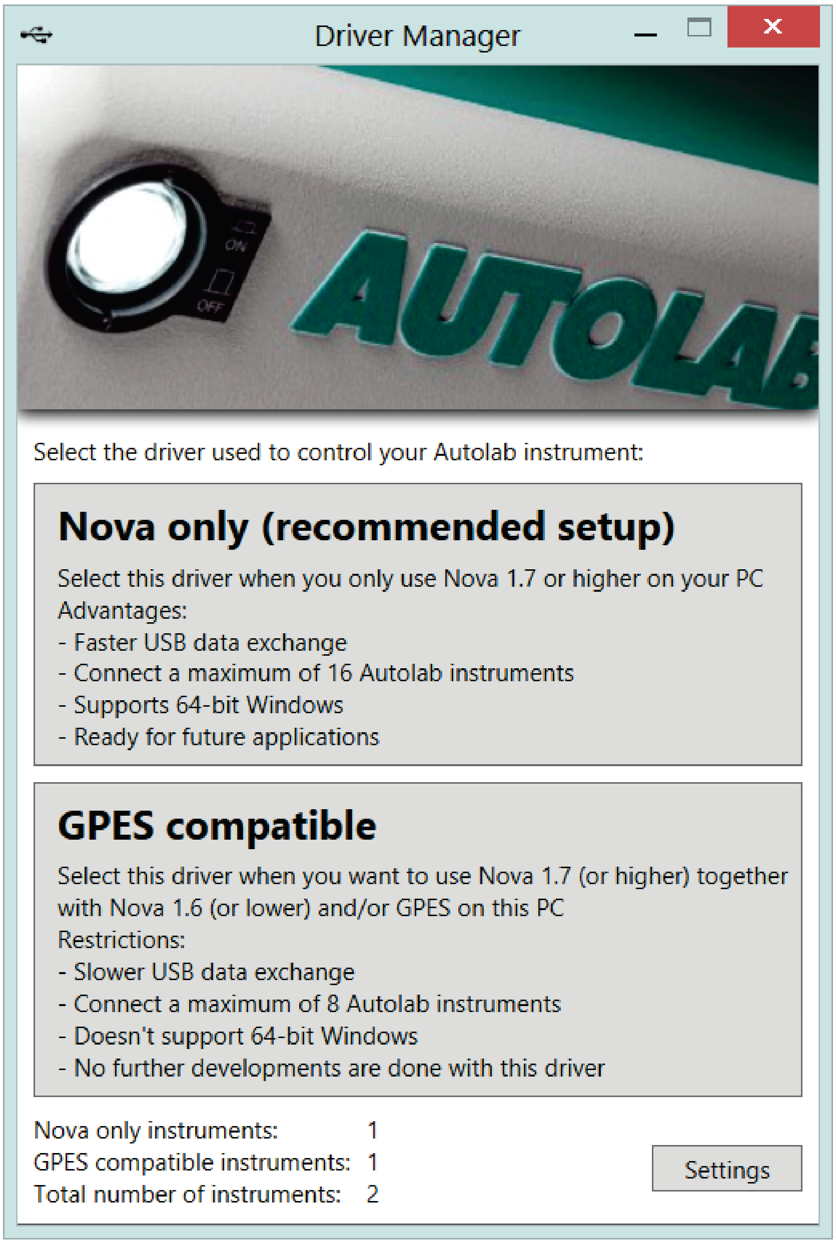


Figure 1.15 – Using the Driver Manager in combination with multiple instruments will update the driver for all the instruments

Clicking either one of the two buttons in the Driver Manager will update the driver, for both instruments, to NOVA only or GPES compatible (depending on the selected driver). In Figure 1.16 all the connected instruments have been updated to NOVA only driver.

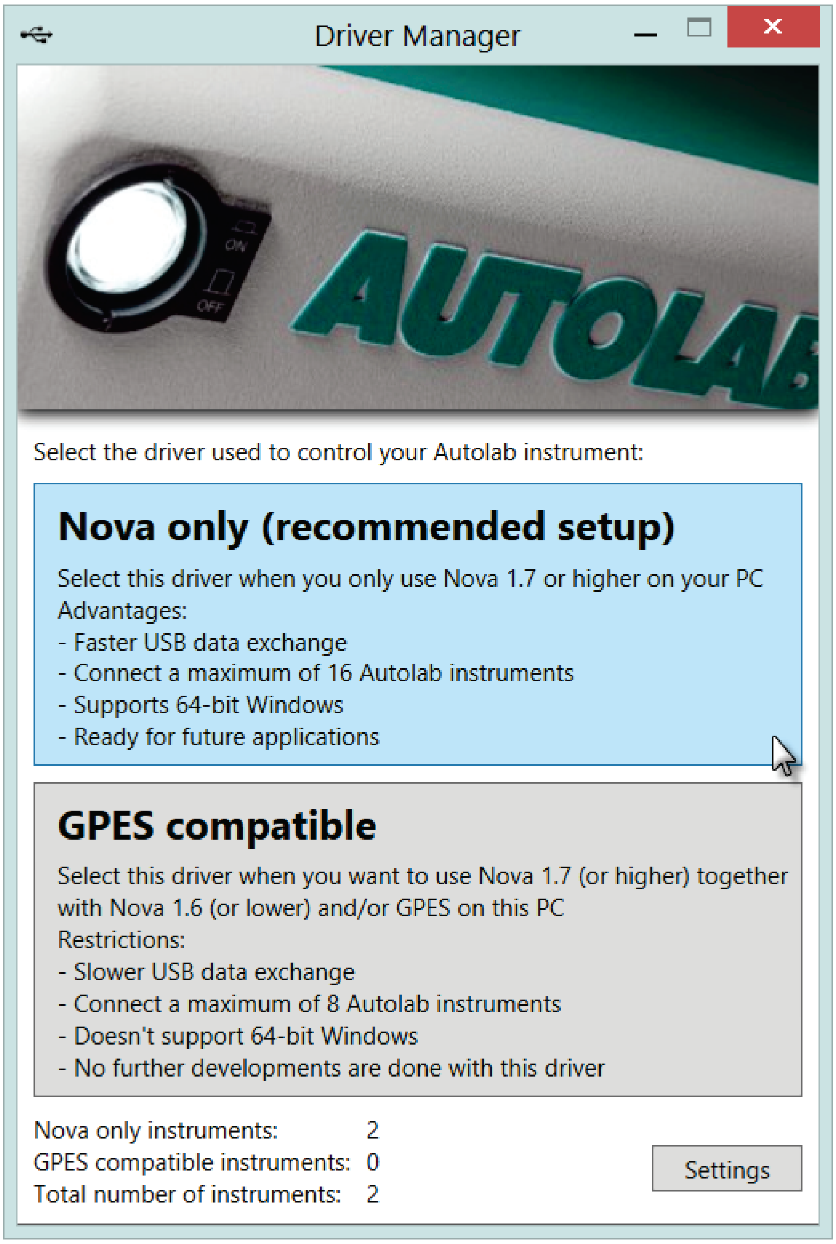


Figure 1.16 – Updating the driver for all the connected instruments

Note

When more than 8 instruments are connected to the same computer through the GPES compatible driver, NOVA will initialize the first 8 instruments and will provide a connection error message in the user log for the remaining instruments (see Figure 1.17).

User log message	Time	Date
All USB addresses are in use. Try using latest driver.	2:14:42 PM	5/23/2011
All USB addresses are in use. Try using latest driver.	2:14:43 PM	5/23/2011
All USB addresses are in use. Try using latest driver.	2:14:43 PM	5/23/2011
All USB addresses are in use. Try using latest driver.	2:14:43 PM	5/23/2011
Autolab/USB connected (µ3AUT70530)	2:14:47 PM	5/23/2011
Autolab/USB connected (µ3AUT70538)	2:14:48 PM	5/23/2011
Autolab/USB connected (µ3AUT70532)	2:14:48 PM	5/23/2011
Autolab/USB connected (µ3AUT70534)	2:14:48 PM	5/23/2011
Autolab/USB connected (AUT83549)	2:14:49 PM	5/23/2011
Autolab/USB connected (AUT83548)	2:14:49 PM	5/23/2011
Autolab/USB connected (AUT83547)	2:14:50 PM	5/23/2011
Autolab/USB connected (AUT83543)	2:14:52 PM	5/23/2011

Figure 1.17 - Error messages are provided when more than 8 instruments are connected to the same computer using the GPES compatible driver

The available instruments will be selected randomly depending on the initialization speed of each Autolab. The excess instruments will not be available for measurements.

Note

More information on the control of multiple instruments in NOVA can be found in the Multi Autolab tutorial, available from the Help menu.

1.3 – Connection to the instrument(s)

When the installation of Nova is finished, start the software by double clicking on the Nova shortcut located on the desktop or by clicking the Nova shortcut located in the Start menu (Start – All Programs – Autolab – Nova) or by using the shortcut tile on the Windows 8 Menu (see Figure 1.18).

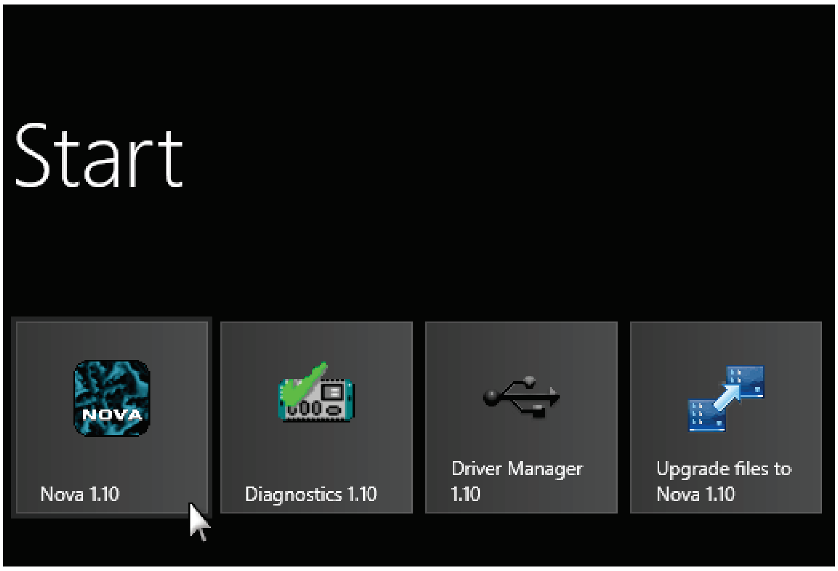


Figure 1.18 – Use the shortcut tile to start NOVA

The software will start and will initiate communication with all the connected instruments (see Figure 1.19).

Note

The upload dialog indicates the USB driver used to control the instrument (GPES for the GPES compatible driver and NOVA for the Nova only driver). Please refer to Section 1.2.5 for more information on the two drivers that can be used to control the Autolab.

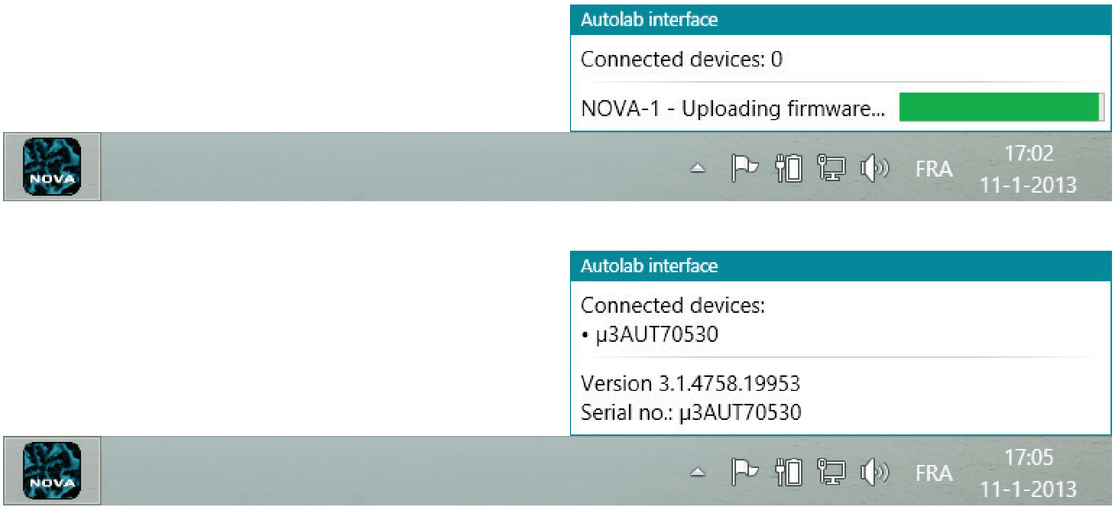


Figure 1.19 – Autolab initialization (top: NOVA only driver in use, bottom: finished initialization)

The initialization can take a few seconds. When it is completed, the serial number of the connected instrument should be displayed, together with the version of the control software (see Figure 1.19).

During the initialization of the instruments, Nova will try to automatically configure each device by detecting the installed modules and type of instrument. This automatic configuration will be triggered whenever an instrument is connected for the very first time. The information about this process is provided in the User log, after initialization (see Figure 1.20).

User log message	Time	Date	Command
Created hardware setup for µ3AUT70530	1:47:41 PM	5/24/2011	-
Autolab/USB connected (µ3AUT70530)	1:47:42 PM	5/24/2011	-

Figure 1.20 – Nova creates the hardware setup automatically for instruments connected the first time

Note

Not all the modules and instruments can be detected automatically. It is always recommended to check the hardware setup after initialization to verify configuration (see Section 1.3.3).

Note

If the computer is connected to the internet, NOVA will automatically check if a new version is available for download on the Metrohm Autolab website.

1.3.1 – Connection and identification of individual instruments

Individual instruments[4] connected to the computer are identified by a unique serial number after the initialization process. Depending on the type of instrument and the configuration, several identification strategies can be encountered.

Instruments with serial number beginning with AUT9 or with µ2AUT7, connected through an external USB interface, are identified by the serial number of the interface, USB7XXXX. Instruments with an internal USB interface, or instruments with serial number beginning with AUT7 connected through an external USB interface, are identified by their own serial number.

Table 1.1 shows an overview of different situations that can be encountered during the initialization of an instrument.

Instrument serial number	USB serial number	Identified as
AUT960512	USB70128	USB70128
AUT71024	USB70256	AUT71024
AUT72048	Internal	AUT72048
AUT84096	Internal	AUT84096
µ2AUT70256	USB70512	USB70512
µ3AUT70384	Internal	µ3AUT70384
AUT40064	Internal	AUT40064
AUT50450	Internal	AUT50450

Table 1.1 – Autolab and USB interface serial number identification examples

[4] This does not apply to the Multi Autolab cabinet, see Section 1.3.2 for more information.

1.3.2 – Connection and identification of the Multi Autolab

M101 modules installed in a Multi Autolab cabinet[5] connected to the computer are identified by a unique composite serial number after the initialization process. The serial number depends on the position of each module in the cabinet, as shown in Figure 1.21.

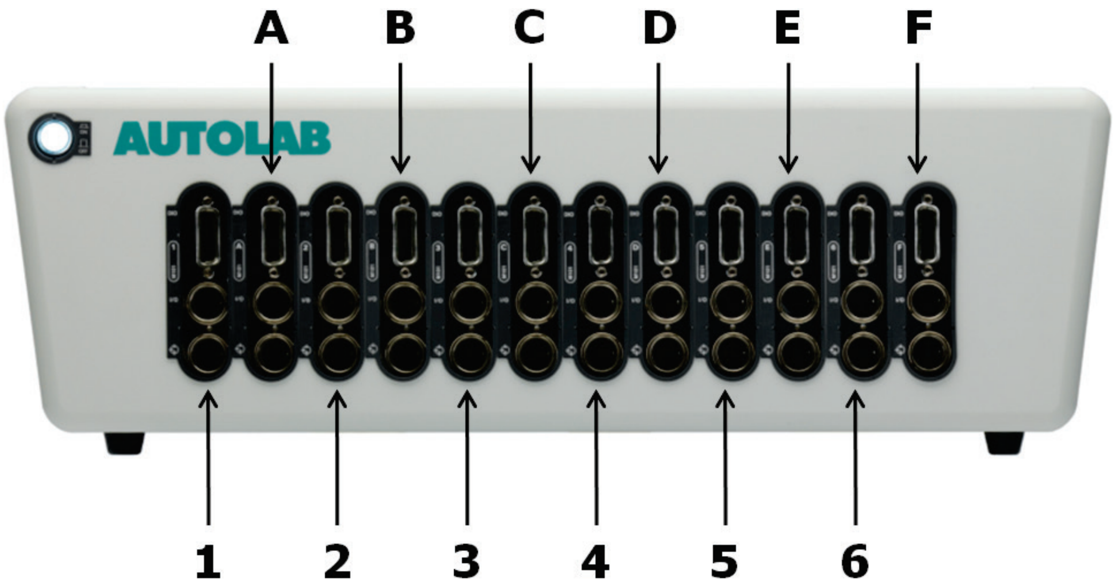


Figure 1.21 – Overview of the Multi Autolab with M101 modules (the module bay labels are indicated by the arrows)

Positions 1 to 6 are known as Parent positions. Positions A to F are daughter positions. Each M101 module in the cabinet is identified by a unique serial number defined by the position of the module and the serial number of the Multi Autolab cabinet[6] (see Table 1.2).

Instrument serial number	USB serial number	Identified as
MAC80001	Internal	MAC80001#1 – 6 (Parent positions)
MAC80001	Internal	MAC80001#A – F (Daughter positions)

Table 1.2 – Multi Autolab serial number identification example

[5] This applies to the Multi Autolab cabinet only, see Section 1.3.1 for more information on the other instruments.
[6] Please refer to the Multi Autolab tutorial, available from the Help menu, for more information.

1.3.3 – Hardware setup

After the software has started, you should see the following screen, which is called the *Setup view* (see Figure 1.22).

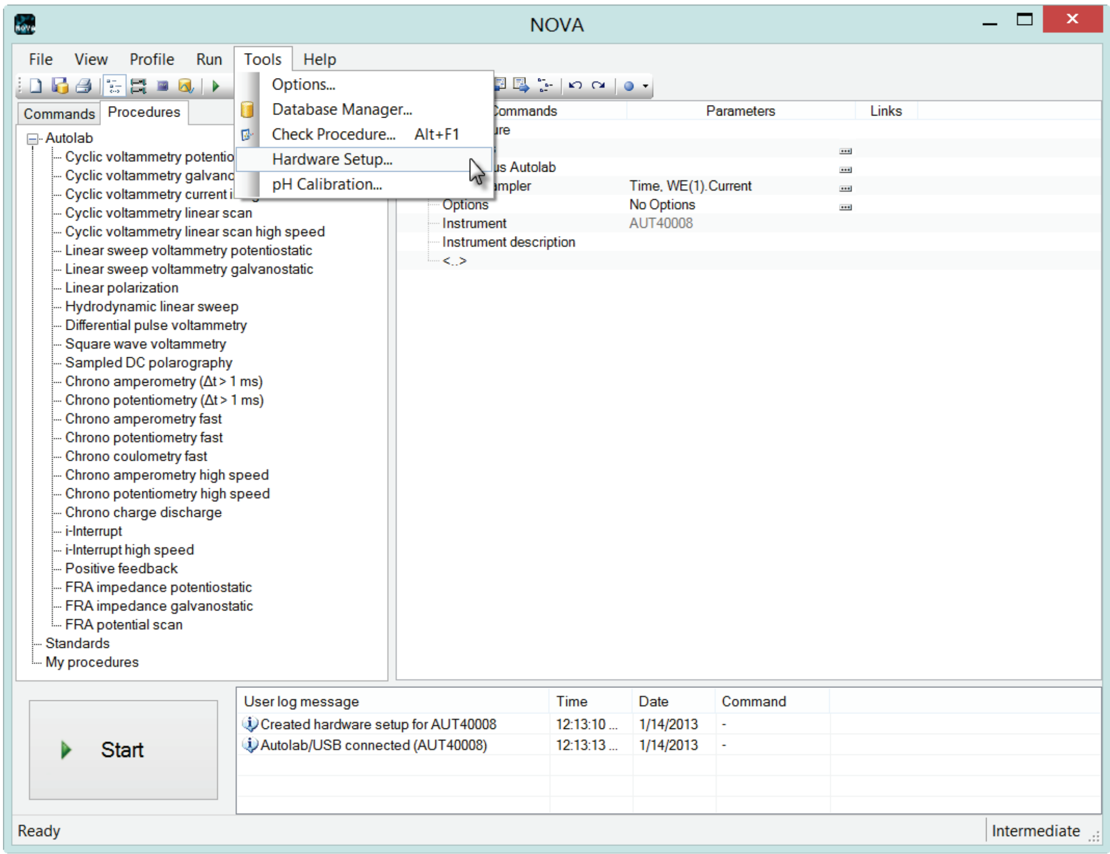


Figure 1.22 – The Setup view of Nova

Locate the **Tools** menu in the toolbar and select the *Hardware setup* from the menu (see Figure 1.22). This will open the Hardware setup window. Check the boxes that correspond to your hardware configuration (see Figure 1.23).

Note

This version of Nova supports all the Autolab instruments (except the µAutolab type I and the PSTAT10) with a USB interface, either internal or through a USB interface box. All the Autolab modules are supported, except the ADC124, DAC124, DAC168 and the first generation FRA.

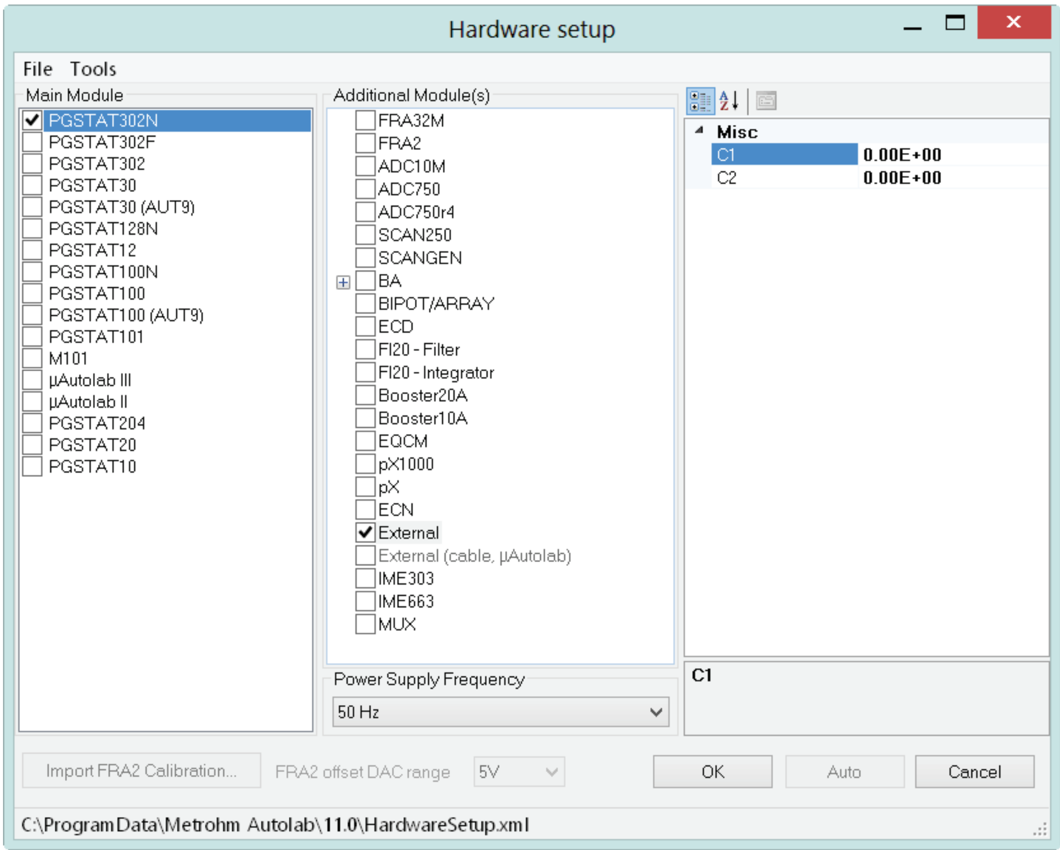


Figure 1.23 – The hardware setup in Nova

Note

Adjust the **Power Supply Frequency** according to your regional settings (50 Hz, 60 Hz).

Click the OK button to close the hardware setup. You will be prompted to confirm the hardware setup (see Figure 1.24).

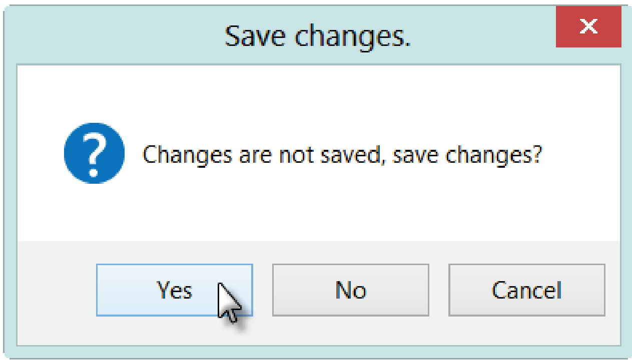


Figure 1.24 – Confirmation of the hardware setup

Note

The hardware setup is saved on the computer using the identifying serial number of the instrument. This hardware configuration will be used automatically whenever the instrument is connected to the computer.

1.4 – FRA2 calibration file

In order to perform electrochemical impedance spectroscopy measurements, the FRA2 module must be installed and the hardware setup in NOVA must be setup accordingly (see Figure 1.23).

Each FRA2 module is calibrated in order to operate correctly inside the Autolab instrument. Before the FRA2 can be used for impedance measurements, the calibration file must be added to the hardware configuration in NOVA.

Note

When NOVA is installed from the CD delivered with a new instrument, the FRA2 calibration file is copied onto the computer automatically, if applicable. This also applies when upgrading an existing NOVA version installed on the computer.

If the FRA2 calibration data is missing, a **warning** message will be displayed in the user log after starting NOVA (see Figure 1.25).

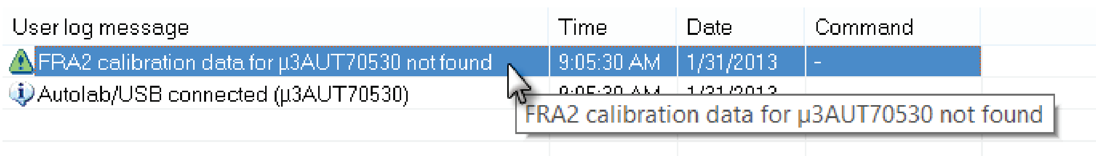


Figure 1.25 – A warning is displayed in the user log when the FRA calibration file is missing

In this case, the FRA2 calibration file must be imported manually. This file (*fra2cal.ini)* can be found in two different locations:

- If the GPES/FRA software is already installed on the computer, the fra2cal.ini file can be found in the C:\autolab folder.
- Alternatively, the fra2cal.ini file can be found on the GPES/FRA 4.9 installation CD matching the serial number of the instrument[7], in the D:\install\disk1 folder.

Warning

If the *fra2cal.ini* file cannot be located, contact your local distributor (serial number of the instrument required).

To import the FRA2 calibration file, select the Hardware setup from the Tools menu. In the Hardware setup window, click the Import FRA2 Calibration... button and locate the file *fra2cal.ini* (see Figure 1.26). Browse to the folder containing the calibration file and click the Open button to load the file.

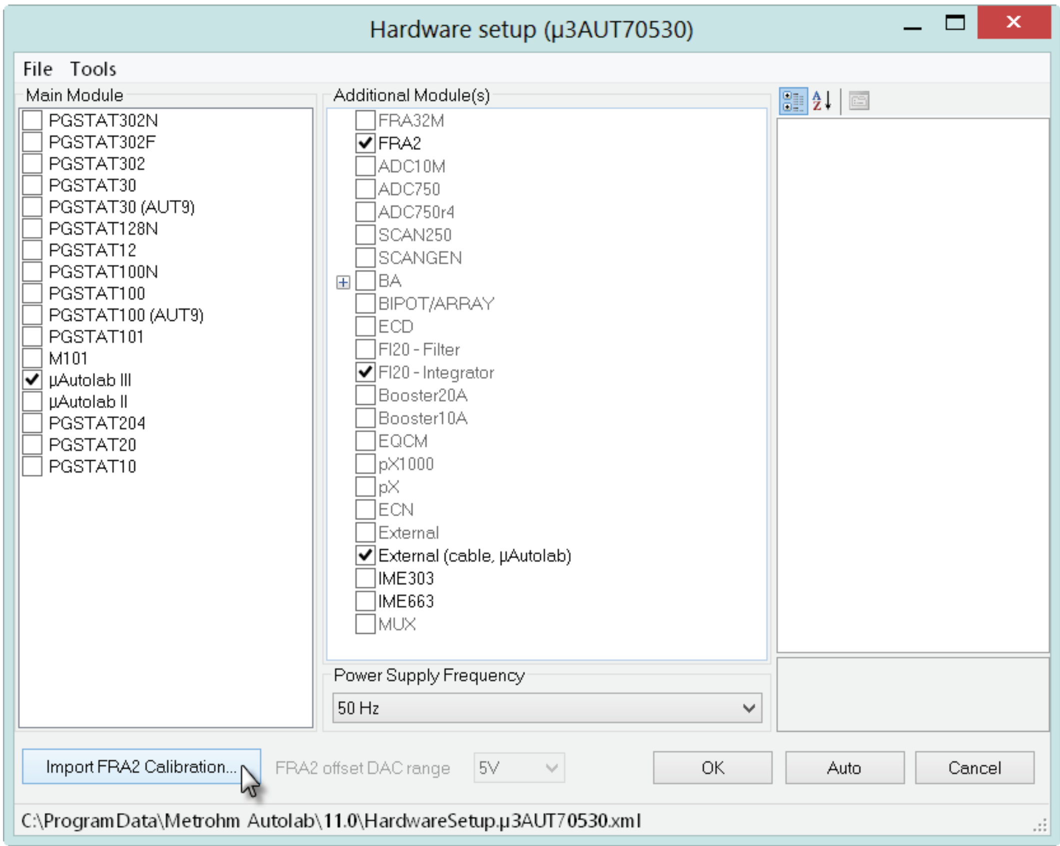


Figure 1.26 – Import the FRA2 calibration file

[7] The serial number of the instrument can be found on label(s) attached to the cell cables or on the back panel of the instrument.

You will be prompted to define the type of instrument for which the fra2cal.ini file is intended (see Figure 1.27).

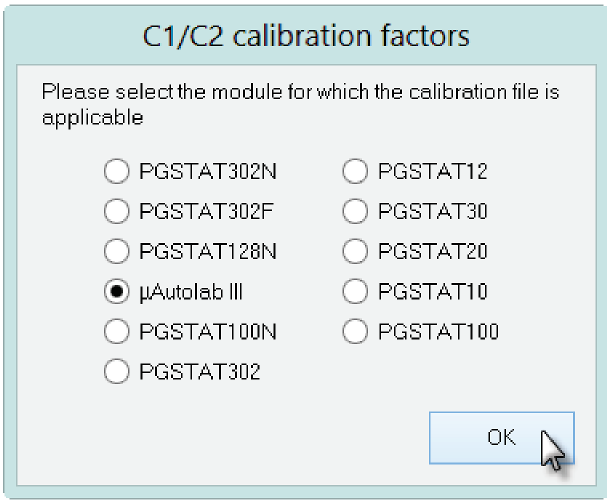


Figure 1.27 – Selecting the instrument type for the fra2cal.ini file

Click the OK button to confirm the selection of the instrument[8] and the OK button in the Nova options window to complete the installation of the FRA2 module calibration file.

Note

If a calibration file was previously imported in Nova, an overwrite warning will be displayed. Click the Yes button to confirm the replacement of the file (see Figure 1.28).

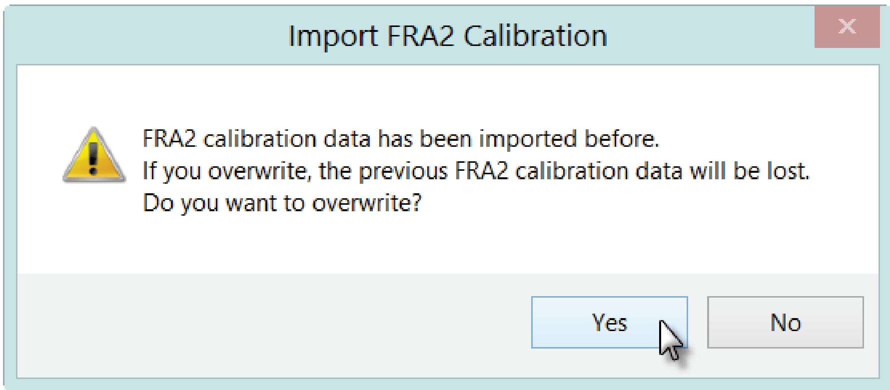


Figure 1.28 – Replacement of a previously defined fra2cal.ini file

[8] See the front panel of the instrument.

Note

The FRA2 calibration file is saved in the hardware setup file of the connected instrument. This calibration data will be automatically whenever the instrument is connected to the computer.

Warning

Depending of the type of FRA2 module, the **FRA offset DAC range** needs to be adjusted to the correct value. FRA2 modules labeled FRA2 V10 on the front panel must be set to **10V** offset DAC range. FRA2 modules labeled FRA2 on the front panel must be set to **5V** (see Figure 1.36). This does not apply to FRA2 modules installed in the μAutolab type III, for which this field is greyed out.

Note

Some FRA2 modules, originally fitted with a **5V** range have been modified to the **10V** range for special applications.

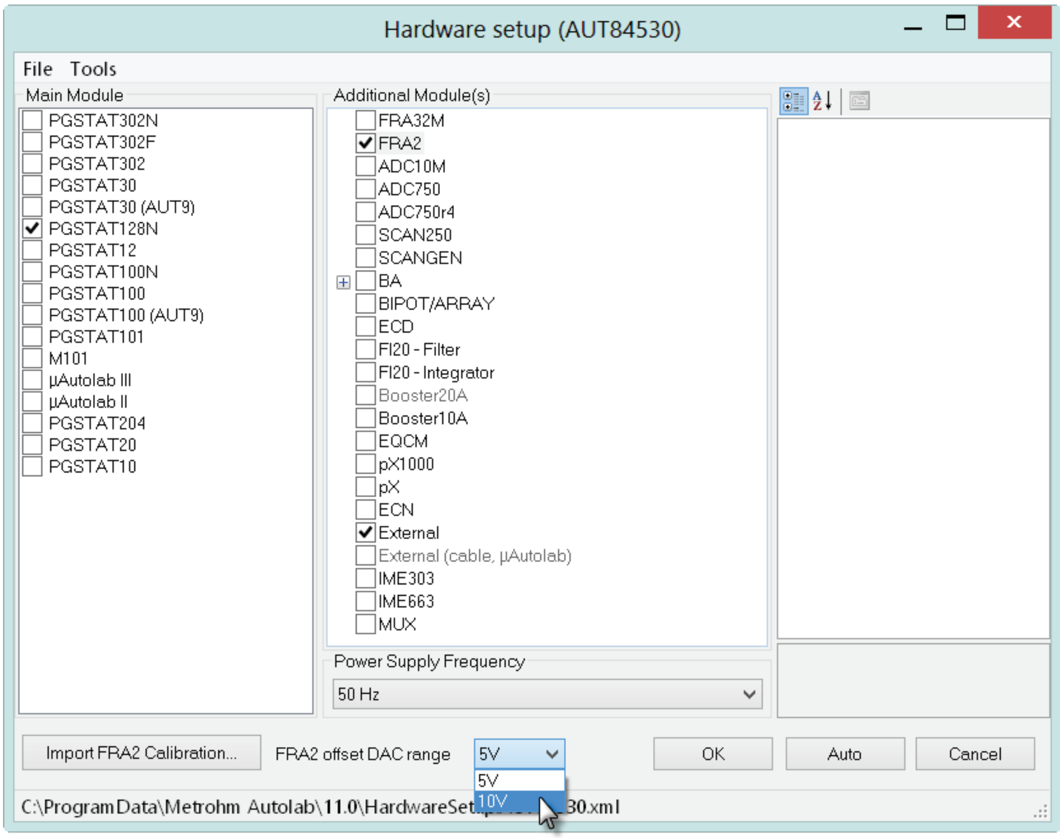


Figure 1.29 – Adjusting the FRA offset DAC range

Warning

For the FRA2 module make sure that the **FRA2 offset DAC range** property is set properly in the hardware setup. For FRA2 modules, the correct value is 5 V. For FRA2.V10 modules, the correct value is 10 V. Failure to set this value properly may result in faulty data at frequencies of 25 Hz and lower (refer to front panel labels of the FRA2 module on the instrument).

1.5 – Diagnostics

Nova includes a diagnostics tool that can be used to test the Autolab instrument. This tool is provided as a standalone application and can be accessed from the start menu, in the Autolab group (Start menu – All programs – Autolab – Tools).

The diagnostics tool can be used to troubleshoot an instrument or perform individual tests to verify the correct operation of the instrument. Depending on the instrument type, the following items are required:

- **μAutolab type II, μAutolab type III and μAutolab type III/FRA2:** the standard Autolab dummy cell. For the diagnostics test, the circuit (a) is used.
- **PGSTAT101 and M101 module:** the internal dummy cell is used during the test, no additional items are required.
- **PGSTAT204:** the standard Autolab dummy cell. For the diagnostics test, the circuit (a) is used.
- **Other PGSTATs:** the standard Autolab dummy cell and a 50 cm BNC cable. For the diagnostics test, the circuit (a) is used. The BNC cable must be connected between the ADC164 channel 2 and the DAC164 channel 2 on the front panel of the instrument[9].

Note

The PGSTAT302F must be tested in normal mode.

[9] In the case of a PGSTAT with serial number not starting with AUT7 or AUT8, connect the BNC cable between DAC channel 4 and ADC channel 4.

The Diagnostics application supports multiple Autolab instruments. When the application starts it detects all available instruments connected to the computer (see Figure 1.30).

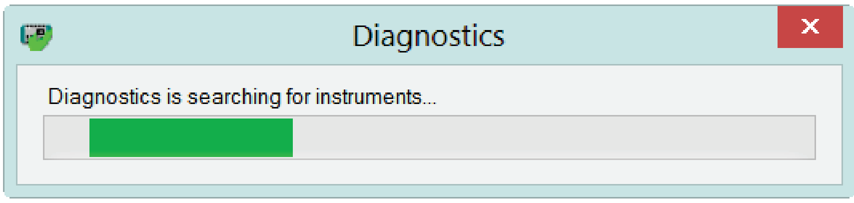


Figure 1.30 – The Diagnostics application automatically scans for all the connected instruments

If more than one instrument is detected, a selection menu is displayed before the Diagnostics starts (see Figure 1.31).

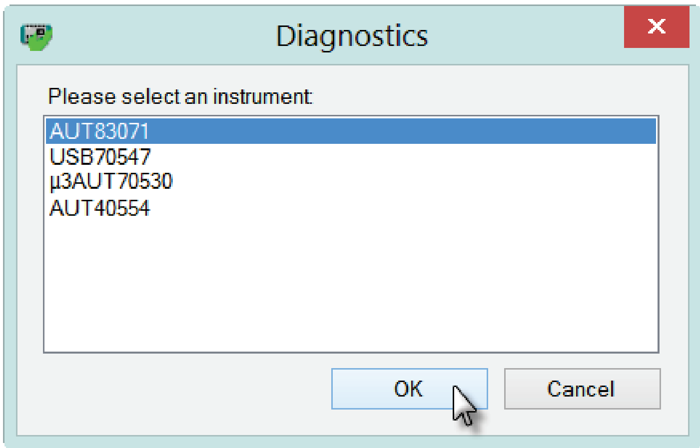


Figure 1.31 – A selection menu is displayed if more than one instrument is detected

The test can only be performed on a single instrument at a time. Select the instrument that needs to be tested and click the OK button to proceed.

When the diagnostics application is started with a Multi Autolab connected, the application will search for the available M101 modules installed in the Multi Autolab and will list the available modules as shown in Figure 1.32.

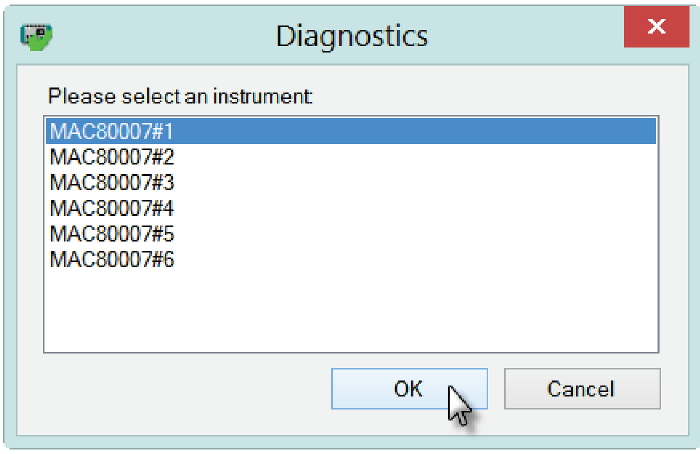


Figure 1.32 – A selection menu identifying the M101 modules by position is displayed when a Multi Autolab is detected by the diagnostics application

The test can only be performed on one channel at a time. Select the M101 module that needs to be tested and click the OK button to proceed.

Note

Instruments with serial number beginning with AUT9 or with µ2AUT7, connected through an external USB interface, are identified by the serial number of the interface, USB7XXXX (see Figure 1.31). Instruments with an internal USB interface, or instruments with serial number beginning with AUT7 connected through an external USB interface, are identified by their own serial number.

When the application is ready, a series of tests can be performed on the selected instrument. In order to perform the tests properly, the hardware setup for the connected instrument must be defined. Select the Hardware option from the Select menu to define or verify the hardware configuration (see Figure 1.33).

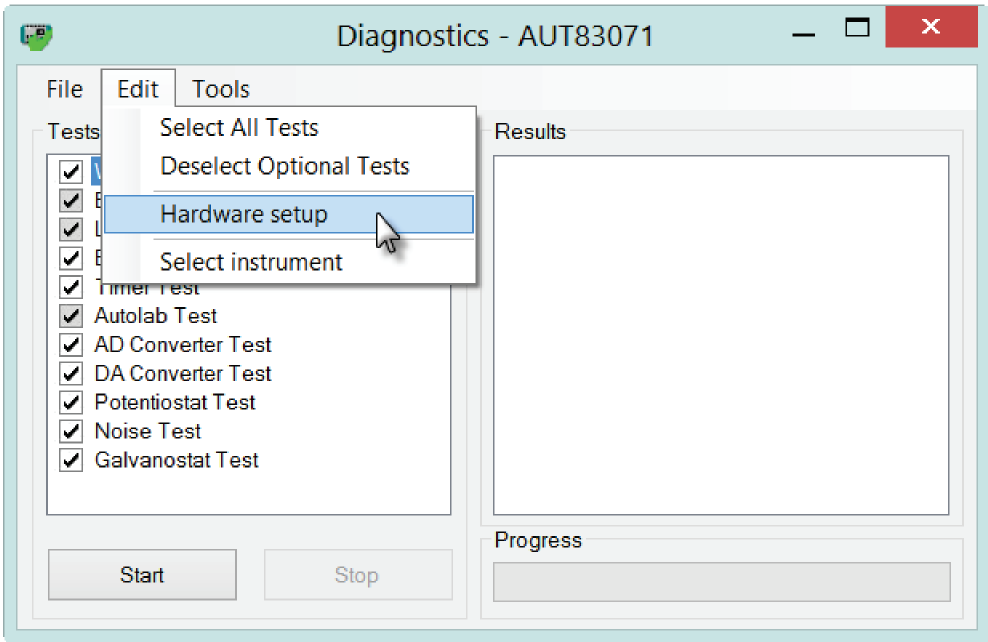


Figure 1.33 – Adjusting the hardware setup for the connected instrument (1/2)

The hardware setup window will be displayed. Adjust the hardware configuration for the connected instrument and press OK to save the changes.

Note

A specific hardware setup file is created and stored on the computer for each instrument.

- If the hardware setup for the connected instrument has already been defined in NOVA or in a previous diagnostics test, the hardware configuration file for the instrument will be automatically recovered and no adjustments will be necessary.
- If no hardware setup file is found for the connected instrument, the default setup is used (default: PGSTAT302N, no additional modules).

Pressing the start button will initiate all the selected tests. A visual reminder will be displayed at the beginning of the test, illustrating the connections required for the test (see Figure 1.34).

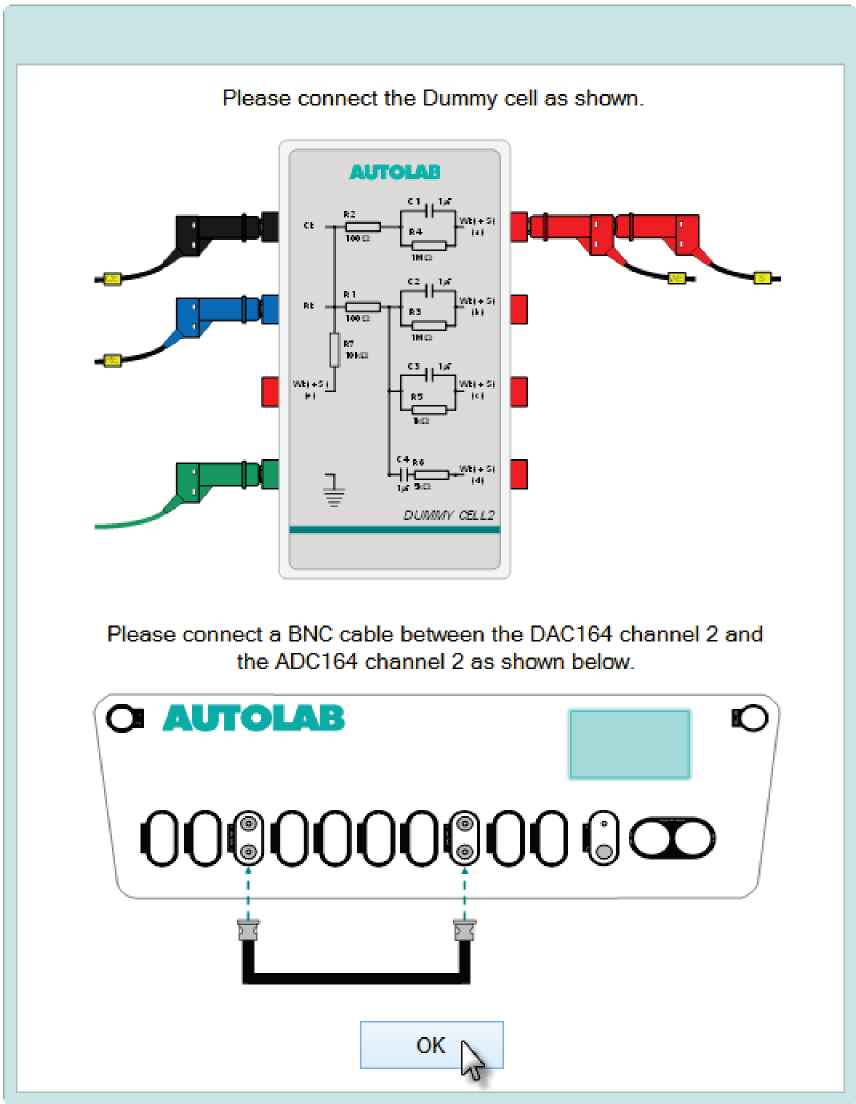


Figure 1.34 – A visual reminder is shown at the beginning of the Diagnostics test

During the test, the progress will be displayed and a successful test will be indicated by a green symbol (see Figure 1.35).

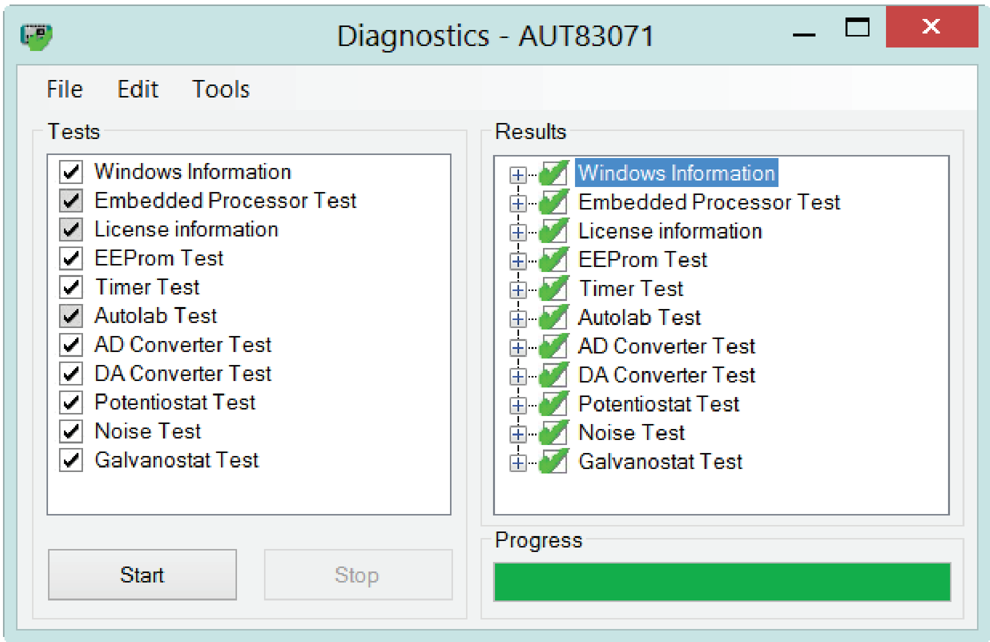


Figure 1.35 – The diagnostics report after all the tests have been performed successfully

If one or more of the tests fails, a red symbol will be used to indicate which test failed and what the problem is. Figure 1.36 shows the output of the diagnostics tool for a failed PSTAT and GSTAT test.

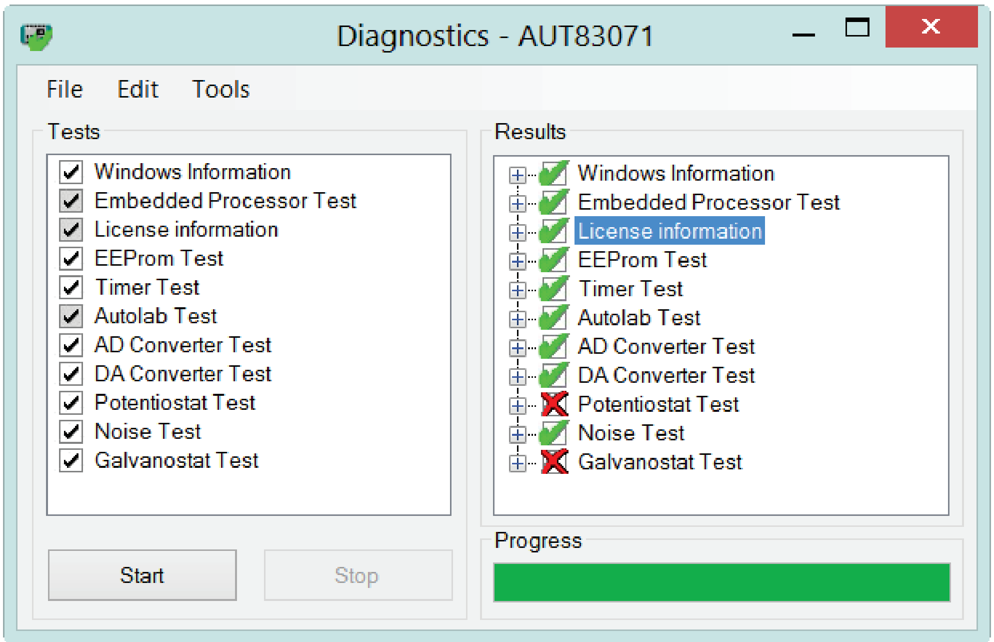


Figure 1.36 – A failed test will be indicated in the diagnostics tool

It is possible to print the test report or to save it as a text file by using the File menu and selecting the appropriate action (see Figure 1.37).

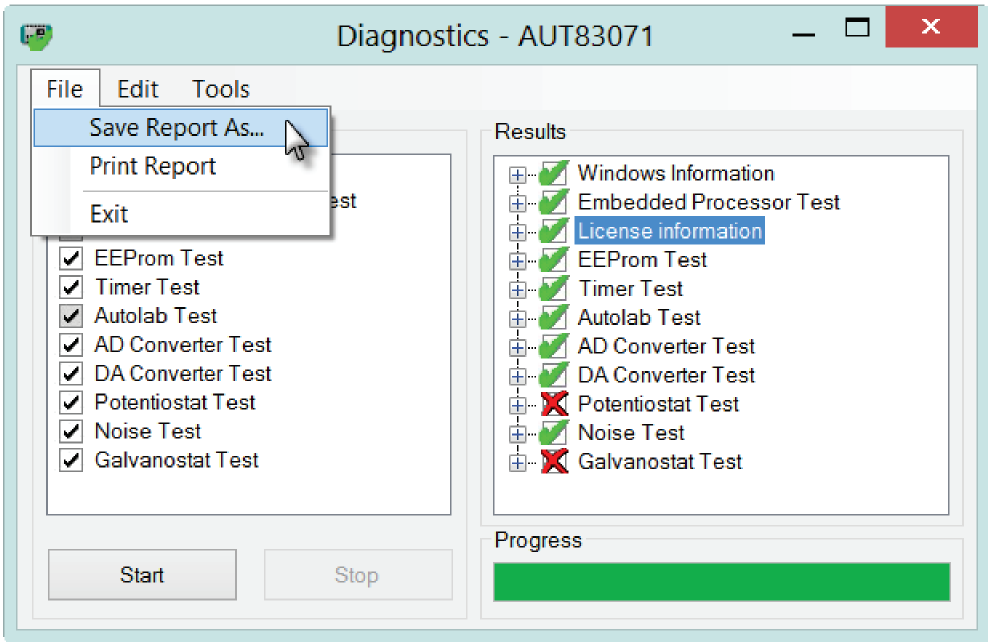


Figure 1.37 – It is possible to save or print the diagnostics report

Note

At the end of the test, it is possible to perform the diagnostics test on another device, if applicable. Use the Select instrument option from the Edit menu to restart the instrument detection (see Figure 1.38). The list of available devices will be displayed after the detection process is finished (see Figure 1.30 and Figure 1.31).

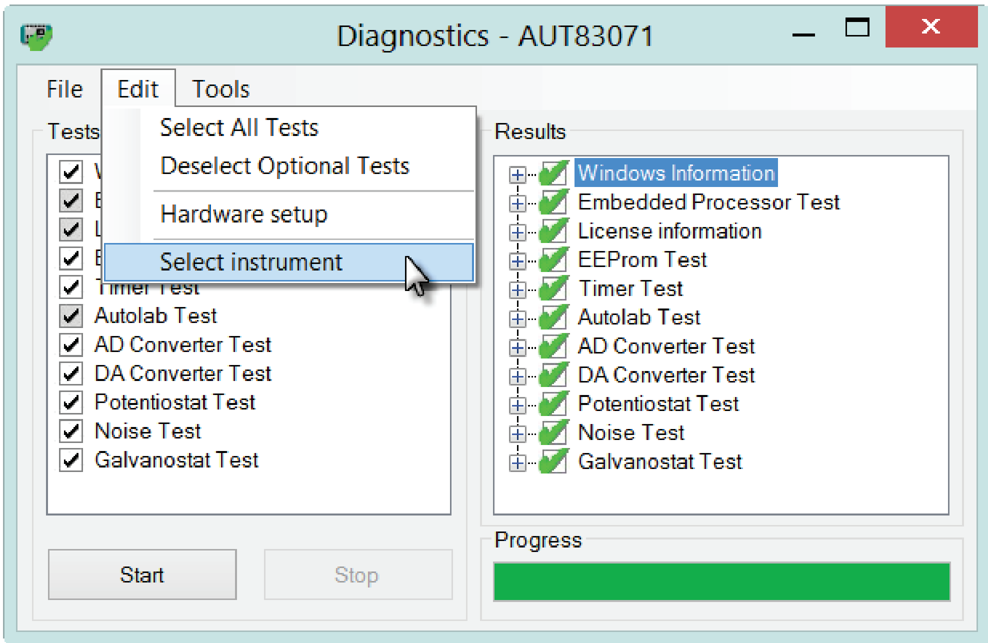


Figure 1.38 – It is possible to restart the instrument detection at the end of the test to diagnose another device

When a FI20-Integrator[10] is specified in the Hardware setup (for instruments with a FI20 module or an on-board integrator), a message will be displayed at the end of the Integrator test (see Figure 1.39).

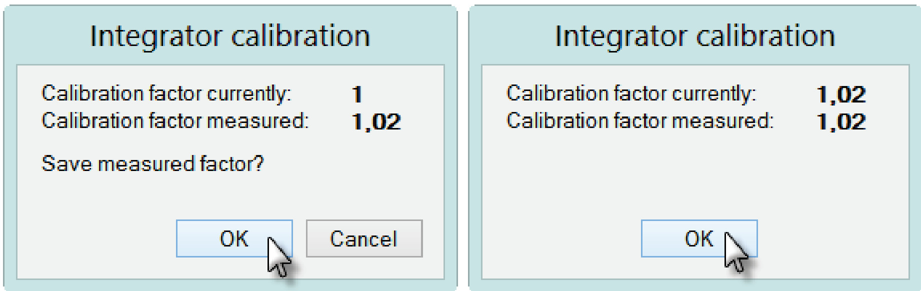


Figure 1.39 – The value of the measured Integrator calibration factor is displayed at the end of the integrator test (left: calibration factor different from stored value, right: calibration factor unchanged)

Click OK to save the measured value in the hardware setup file of the instrument.

[10] The determination of the integrator calibration factor does not replace the full test of the module. Please refer to Sections 1.6.9-1.6.11 or to the Module test document, available from the Help menu for more information on the complete test of the FI20-Integrator.

1.5.1 – Autolab Firmware Update

For some instruments, a firmware update may be required. If this is the case of the connected instrument, a message will be displayed during the Diagnostics test (see Figure 1.40).

During Diagnostics, an update message will be displayed if the outdated firmware is detected. Clicking the Yes button when prompted will silently update the firmware (see Figure 1.40).

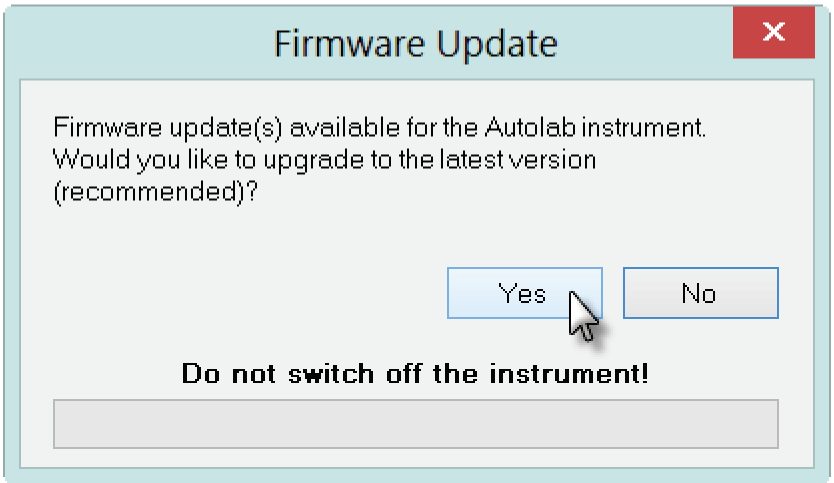


Figure 1.40 – An upgrade message is displayed when the outdated firmware is detected

The firmware update is permanent. The Firmware Update window will close automatically at the end of the update and the diagnostics test will continue.

Important

Do not switch off the instrument or disconnect the instrument during the firmware update since this will damage the instrument.

1.6 – Module test in NOVA

Nova includes a number of procedures designed to verify the basic functionality of the different hardware modules installed in the instrument. These tests can be performed at any time using the Autolab dummy cell[11].

These procedures are located in the *Module test* database located in the *C:\Program Files\Metrohm Autolab\Nova 1.10\Shared DataBases* folder. To use these procedures, define the location of the *Module test* folder as the Standard database, using the Database manager, available from the Tools menu (see Figure 1.41).

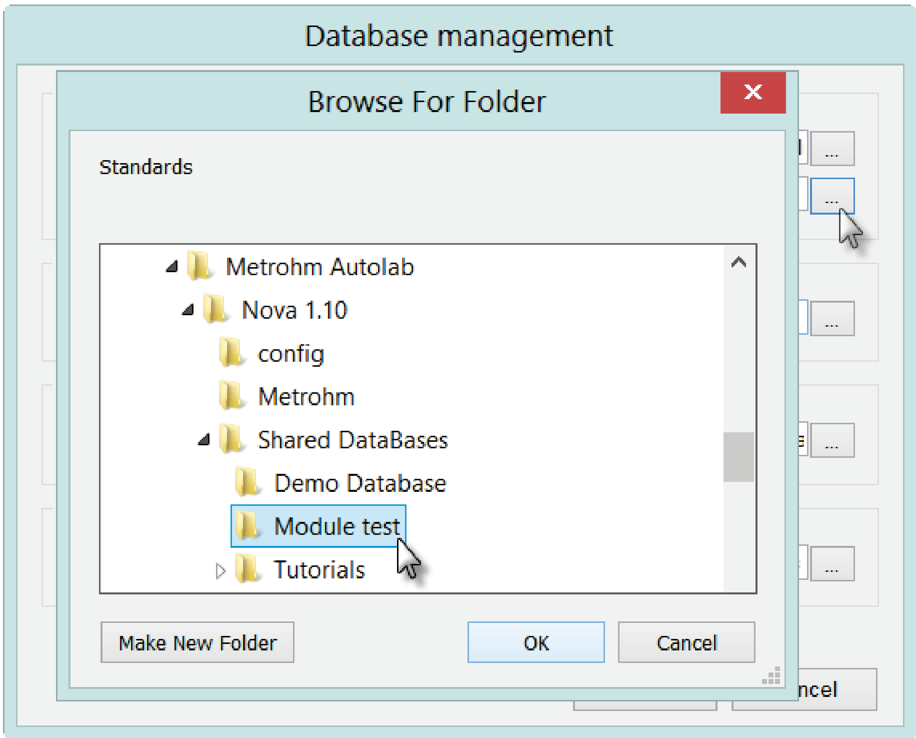


Figure 1.41 – Loading the Module test database

A total of 25 procedures are provided in the *Module test* database (see Figure 1.42).

[11] Except for the Autolab PGSTAT101, the Autolab M101 module and the Autolab EQCM module.

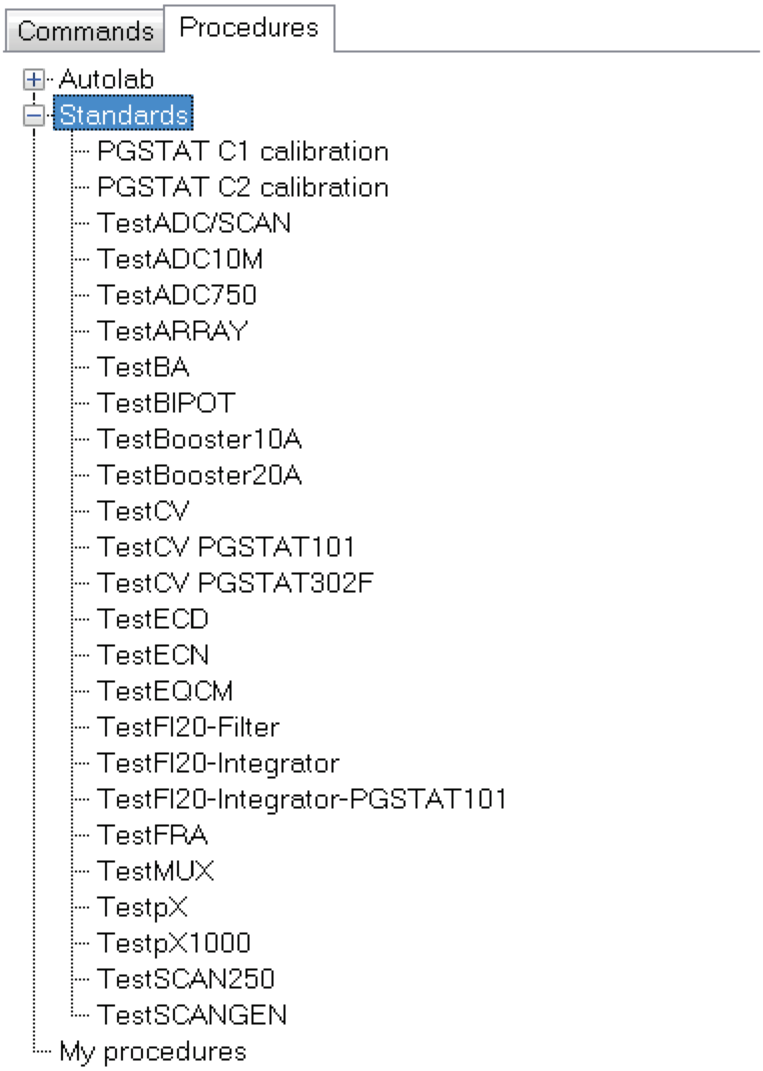


Figure 1.42 – The Module test procedures

The first two procedures (*PGSTAT C1 calibration* and *PGSTAT C2 calibration*) are special procedures used to determine the **C1** and **C2** factors required for the operation of the FRA32M or the FRA2 module in combination with the Autolab. These procedures are intended to be used under the experimental conditions described in the module installation documentation. Please refer to Section 1.6.18 for more information.

The other 23 procedures can be used at any time to test the different hardware modules installed in the instrument.

This section provides a short description of the test procedures included in the Hardware test database.

Note

Make sure that the hardware setup is defined correctly (see Section 1.3).

1.6.1 – Test of the Autolab PGSTAT

1.6.1.1 – Test of the Autolab PGSTAT128N, 302N, 302F (normal mode), 100N, 204 and µAutolab III

This simple test is designed to verify the basic functionality of the potentiostat[12]. It can be used to test all the Autolab PGSTAT instruments **except** the Autolab PGSTAT101, the Autolab M101 potentiostat/galvanostat module[13] and the PGSTAT302F in normal mode[14].

Load the **TestCV** procedure from the Standards database, connect dummy cell (a) and press the start button (see Figure 1.43).

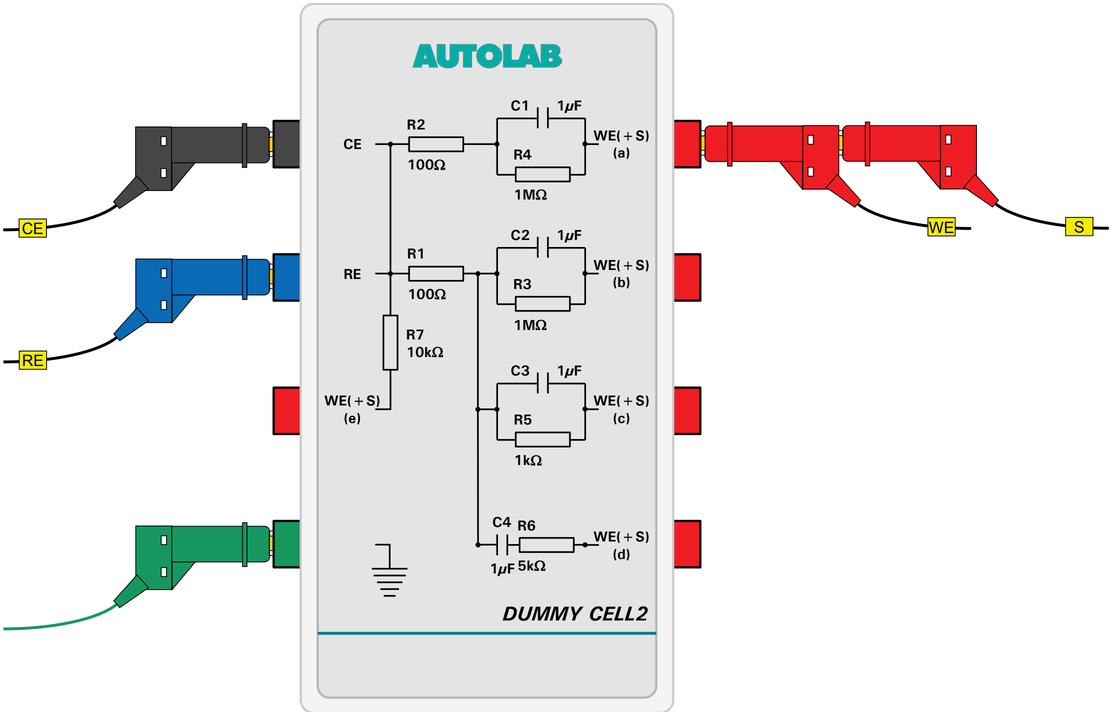


Figure 1.43 – The TestCV procedure requires connection to the dummy cell (a)

[12] This test is also used to test earlier Autolab instruments (PGSTAT10, 20, 12, 30, 302, 100) and the µAutolabII.
[13] A specific test for the PGSTAT101 and the M101 is provided (see section 1.6.1.2).
[14] A specific test for the PGSTAT302F in floating mode is provided (see section 1.6.1.3).

A message will be displayed when the measurement starts (see Figure 1.44).

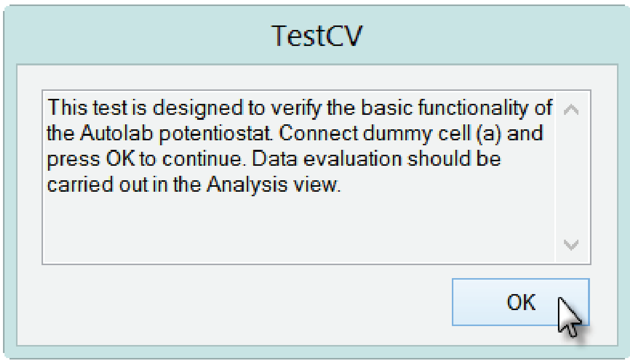


Figure 1.44 – A message is displayed at the beginning of the measurement

The test uses the cyclic voltammetry staircase method and performs a single potential scan starting from 0 V, between 1 V and -1V. At the end of the measurement, switch to the Analysis view and load the data for evaluation.

The data set includes three groups of data points (see Figure 1.45).

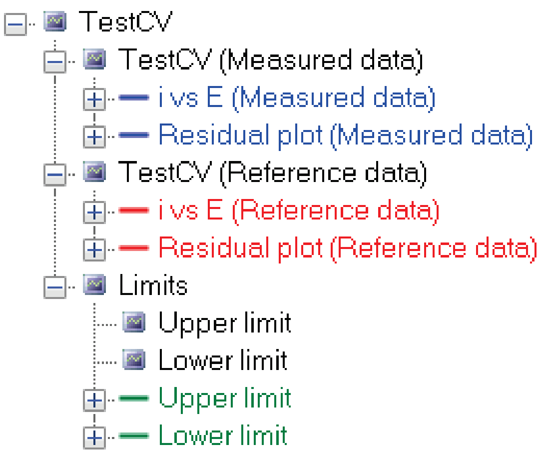


Figure 1.45 – The data obtained with the TestCV procedure

The first group, located under TestCV (Measured data) contains the measured curve and the data after baseline correction (see Figure 1.46).

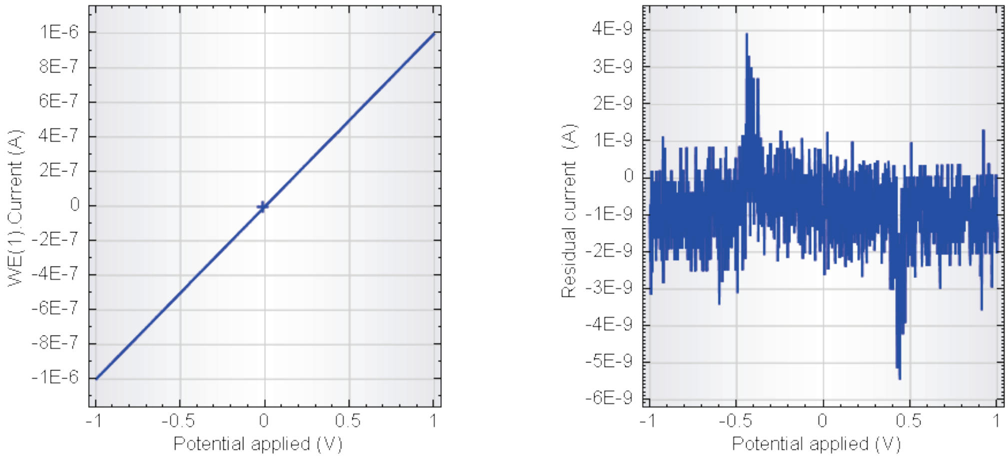


Figure 1.46 – The data points recorded during the TestCV measurement (left) and the data points after linear baseline correction (right)

The difference between the maxima observed in the residual current plot should be < 40 nA.

The second group, located under TestCV (Reference data) contains data from a reference measurement. This data can be used for comparison with the data points obtained during the test. Two reference curves are provided: the i vs E plot and the Residual plot after baseline correction.

The third group, located under Limits, contains the absolute maximum and minimum limit allowed for the residual current calculated from the measured data points.

Figure 1.47 shows an overlay of the residual current calculated from the measured data, the residual current plot provided as reference data and the absolute limits allowed for the residual current.

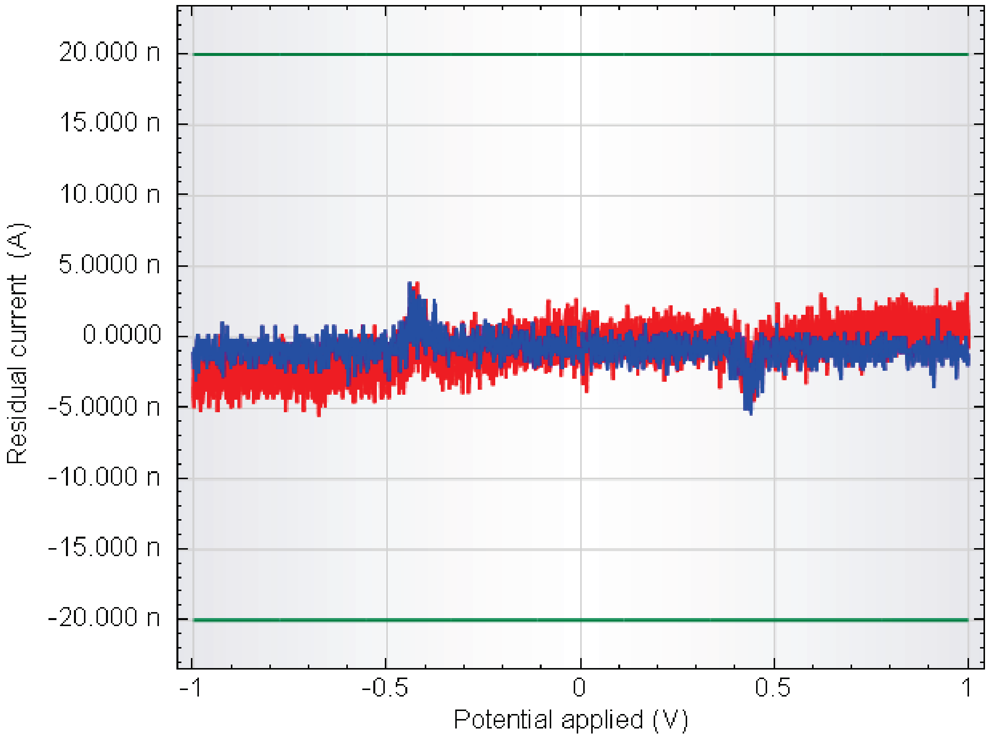


Figure 1.47 – An overlay of the residual current obtained from the measured data (blue curve), the residual current from the reference data (red curve) and the absolute limits (green lines)

1.6.1.2 – Test of the Autolab PGSTAT101 and M101

This simple test is designed to verify the basic functionality of the Autolab PGSTAT101 and the Autolab M101 potentiostat/galvanostat module[15].

Load the **TestCV PGSTAT101** procedure from the Standards database. This test uses the internal dummy cell of the instrument. Connect the CE and the RE electrode leads and the WE and S from the cell cable as shown in Figure 1.48 and press the start button.

[15] For testing the PGSTAT302F in floating mode please refer to section 1.6.1.3. For testing all the other Autolab instruments, please refer to section 1.6.1.1.

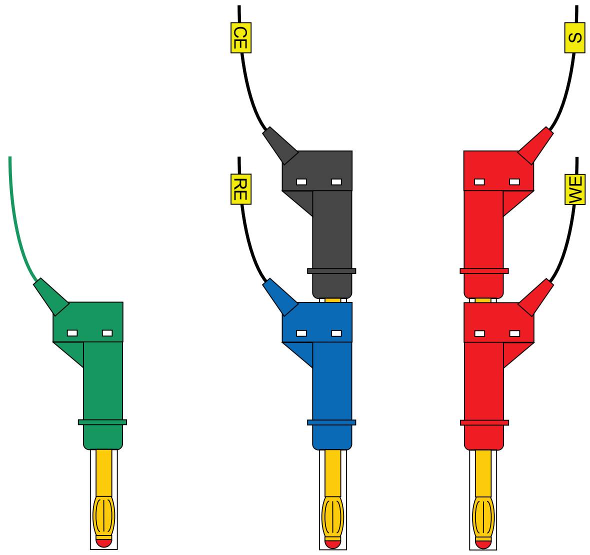


Figure 1.48 – The connections required for the PGSTAT101 test

A warning message, indicating that the internal dummy cell is used, will be shown during validation (see Figure 1.49). This warning is provided as a reminder and the OK button can be clicked to proceed with the measurement.

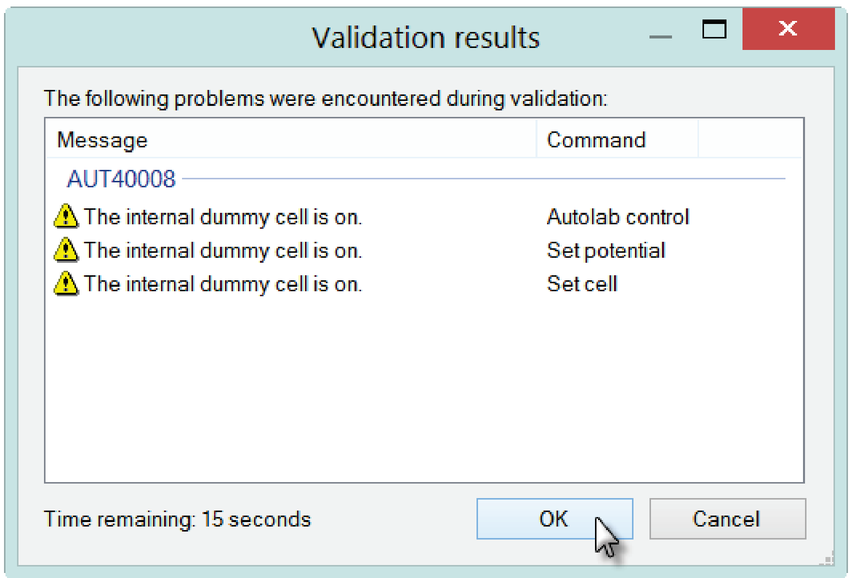


Figure 1.49 – A warning message is shown during validation

A message will be displayed when the measurement starts (see Figure 1.50).

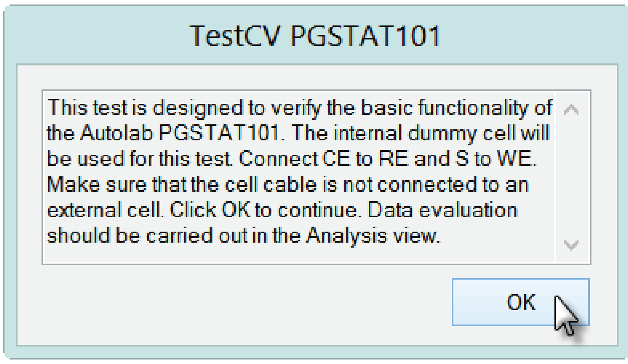


Figure 1.50 – A message is displayed at the beginning of the measurement

The test uses the cyclic voltammetry staircase method and performs a single potential scan starting from 0 V, between 1 V and -1V. At the end of the measurement, switch to the Analysis view and load the data for evaluation.

The data set includes two groups of data points (see Figure 1.51).

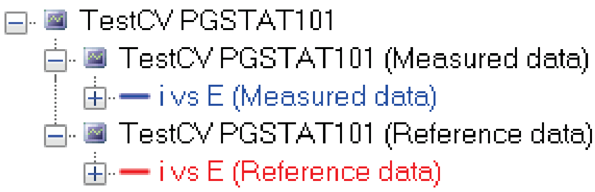


Figure 1.51 – The data obtained with the TestCV PGSTAT101 procedure

The first group contains the measured data points. The other group contains data points from a reference measurement. This data can be used for comparison with the data points obtained during the test.

The measured data should be similar to the reference data provided for comparison, as shown in Figure 1.52.

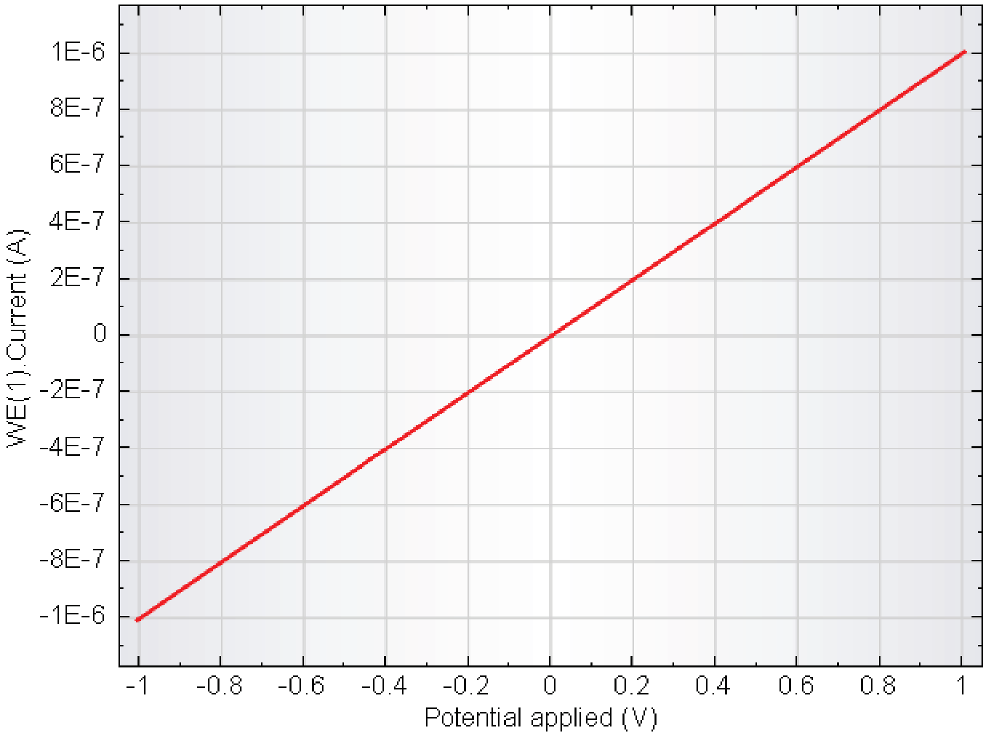


Figure 1.52 – The expected result of the TestCV PGSTAT101 procedure

The test is successful if the measured data can be compared to the reference data.

1.6.1.3 – Test of the Autolab PGSTAT302F in floating mode

This simple test is designed to verify the basic functionality of the Autolab PGSTAT302F in **floating mode** only[16].

Load the **TestCV PGSTAT302F** procedure from the Standards database, connect dummy cell (a) as shown in Figure 1.53 and press the start button.

[16] For testing the PGSTAT101 and the M101 module, please refer to Section 1.6.1.2. For testing all the other Autolab instruments, including the PGSTAT302F in normal mode, please refer to Section 1.6.1.1.

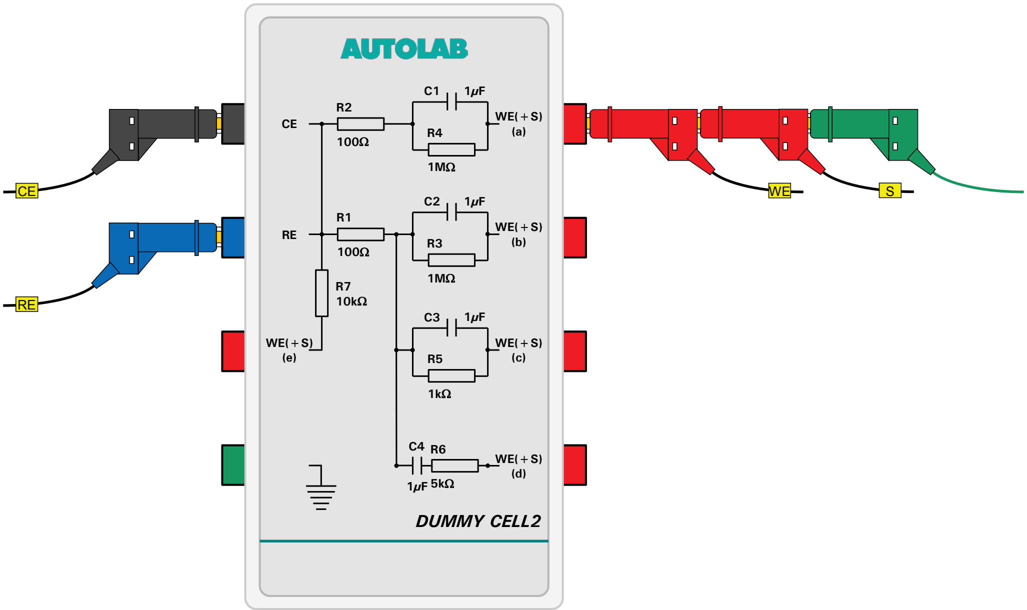


Figure 1.53 – The connections to the dummy cell (a) required to test the PGSTAT302F in floating mode

A message will be displayed when the measurement starts (see Figure 1.54).

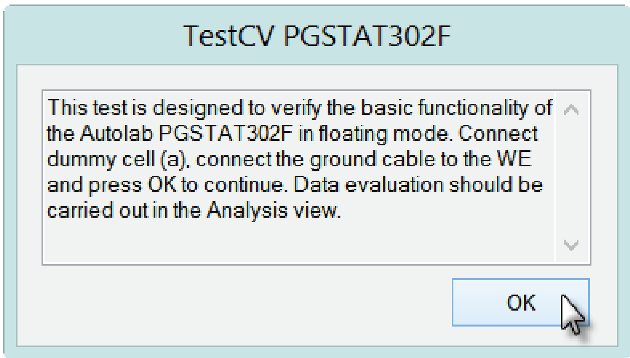


Figure 1.54 – A message is displayed at the beginning of the measurement

The test uses the cyclic voltammetry staircase method and performs a single potential scan starting from 0 V, between 1 V and -1V. At the end of the measurement, switch to the Analysis view and load the data for evaluation.

The data set includes three groups of data points (see Figure 1.55).

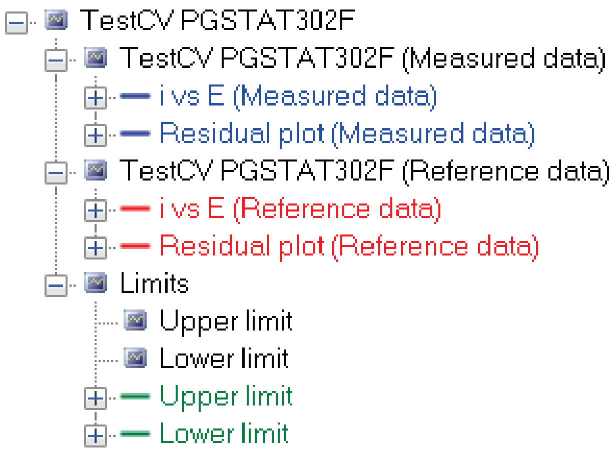


Figure 1.55 – The data obtained with the TestCV procedure

The first group, located under TestCV PGSTAT302F (Measured data) contains the measured curve and the data after baseline correction.

The second group, located under TestCV PGSTAT302F (Reference data) contains data from a reference measurement. This data can be used for comparison with the data points obtained during the test. Two reference curves are provided: the i vs E plot and the Residual plot after baseline correction.

The third group, located under Limits, contains the absolute maximum and minimum limit allowed for the residual current calculated from the measured data points.

Figure 1.56 shows an overlay of the residual current calculated from the measured data, the residual current plot provided as reference data and the absolute limits allowed for the residual current.

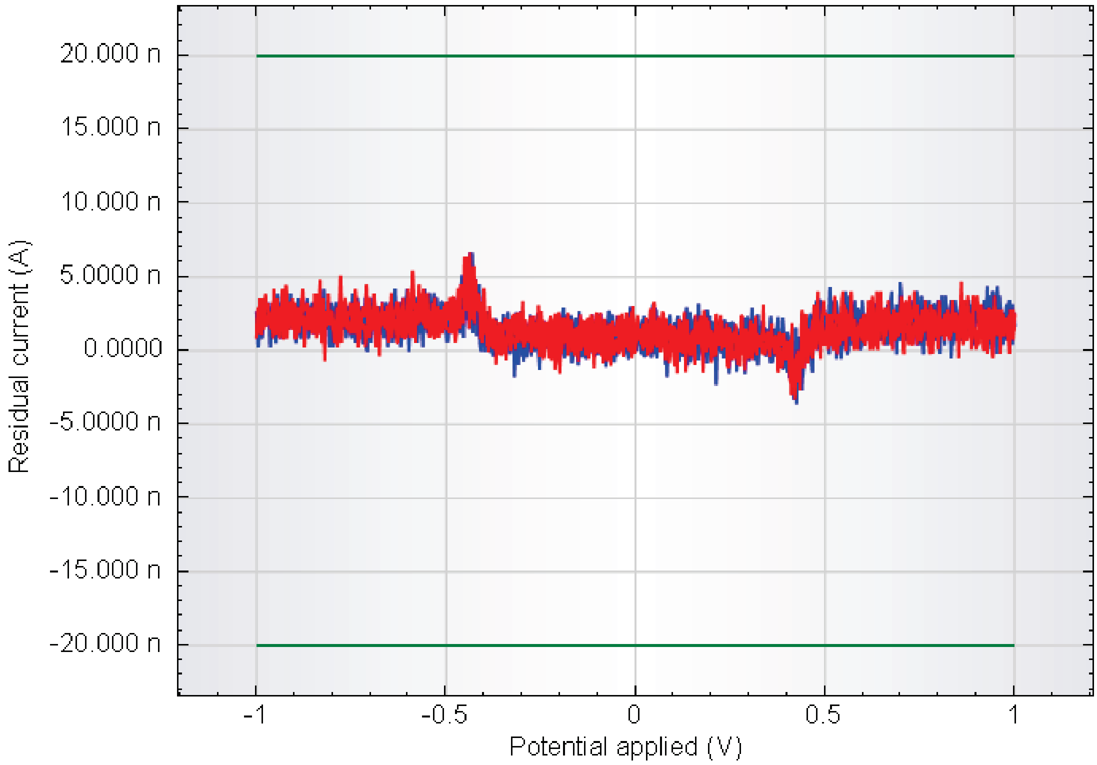


Figure 1.56 – An overlay of the residual current obtained from the measured data (blue curve), the residual current from the reference data (red curve) and the absolute limits (green lines)

1.6.2 – Test of the ADC750 or the ADC10M

Two procedures, TestADC750 and TestADC10M can be used to test the correct functionality of the fast sampling ADC module (ADC750 or ADC10M, respectively).

Load the **TestADC750** or the **TestADC10M** procedure depending on the module to test from the Standards database, connect dummy cell (c) and press the start button.

A message will be displayed when the measurement starts (see Figure 1.57).

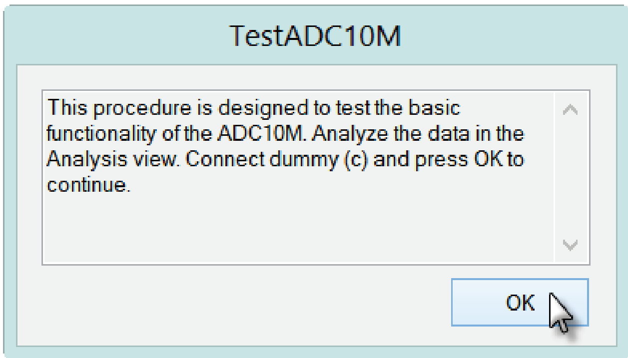


Figure 1.57 – A message is displayed at the beginning of the measurement

Note

No data points can be shown real time during measurements with the fast-sampling ADC module.

The test uses the chrono amperometry high speed method and performs a total of four potential steps. At the end of the measurement, switch to the Analysis view and load the data for evaluation.

The data set includes two groups of data points (see Figure 1.58).

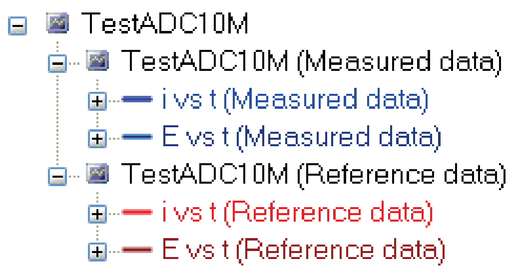


Figure 1.58 – The data obtained with the TestADC10M procedure

Note

The data for the TestADC750 is displayed in a similar way.

The first group, located under TestADC10M (Measured data) contains the measured current and measured potential plotted versus corrected time. The second group contains data from a reference measurement. This data can be used for comparison with the data points obtained during the test.

The measured data should be similar to the reference data provided for comparison as shown in Figure 1.59.

Note

Small deviation can be observed between the measured data points and the reference data because of the tolerance of the capacitance included in the dummy cell (± 5%).

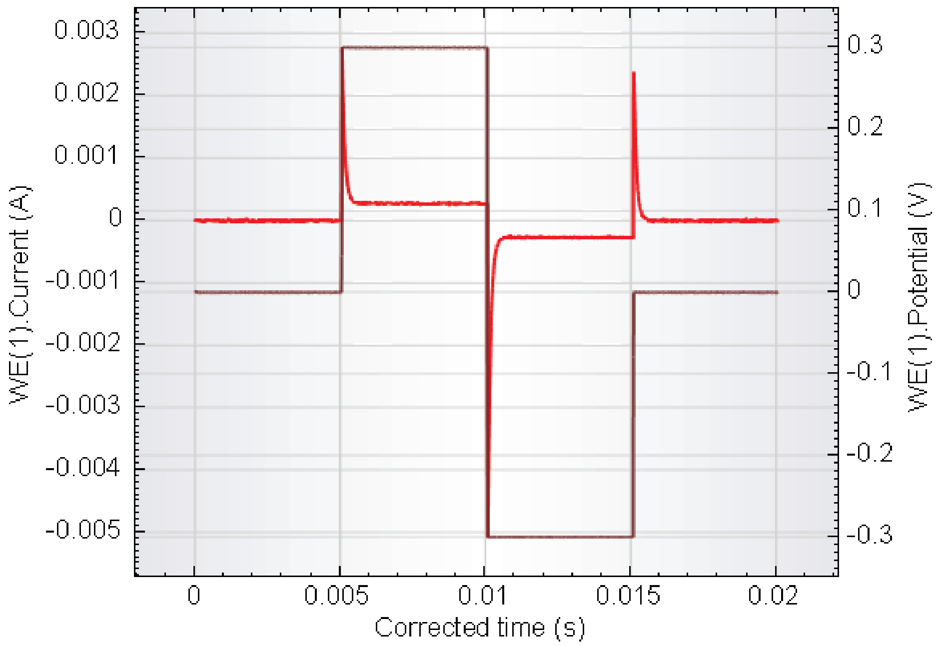


Figure 1.59 – The expected result of the TestADC10M or the TestADC750 procedure (red curve: WE(1).Current, brown curve: WE(1).Potential)

The test is successful if the measured data can be compared to the reference data.

1.6.3 – Test of BA

The TestBA procedure can be used to test the correct functionality of the BA module. The BA module is a dual mode module that works both as a bipotentiostat and as a scanning bipotentiostat.

Load the **TestBA** procedure, connect WE(1) to dummy cell (a) and WE(2) to dummy cell (b) as shown in Figure 1.60 and press the start button.

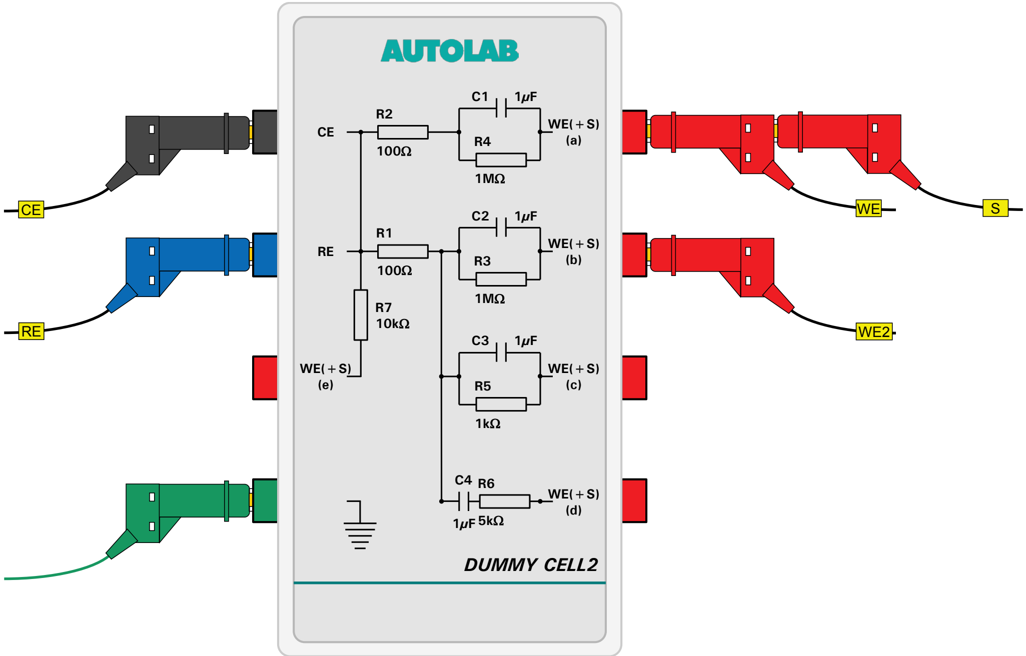


Figure 1.60 – Overview of the connections to the dummy cell required for the TestBA, TestBIPOT and TestARRAY procedures

A message will be displayed when the measurement starts.

Note

Two measurements are performed during the test.

The test uses the cyclic voltammetry staircase method and performs a total of two potential scans. During the first scan, the BA is set to Bipotentiostat mode (potential of WE(2) is expressed relative to the potential of the reference electrode). During the second scan, the BA is set to scanning bipotentiostat mode (potential of WE(2) is expressed relative to the potential of WE(1)). In both measurements, the offset potential used for WE(2) is 1 V.

At the end of the measurement, switch to the Analysis view and load the data for evaluation.

The data set includes four groups of data points (see Figure 1.61).

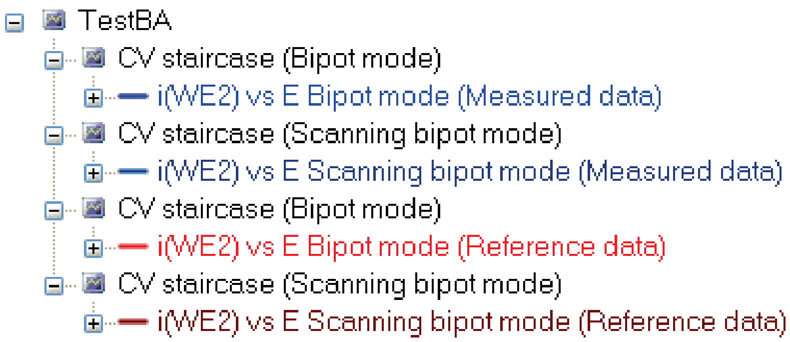


Figure 1.61 – The data obtained with the TestBA procedure

The first two groups contain the measured data points for the WE(2).Current in Bipot mode and in Scanning Bipot mode. The other two groups contain data points for the WE(2).Current from a reference measurement. This data can be used for comparison with the data points obtained during the test.

The measured data should be similar to the reference data provided for comparison as shown in Figure 1.62.

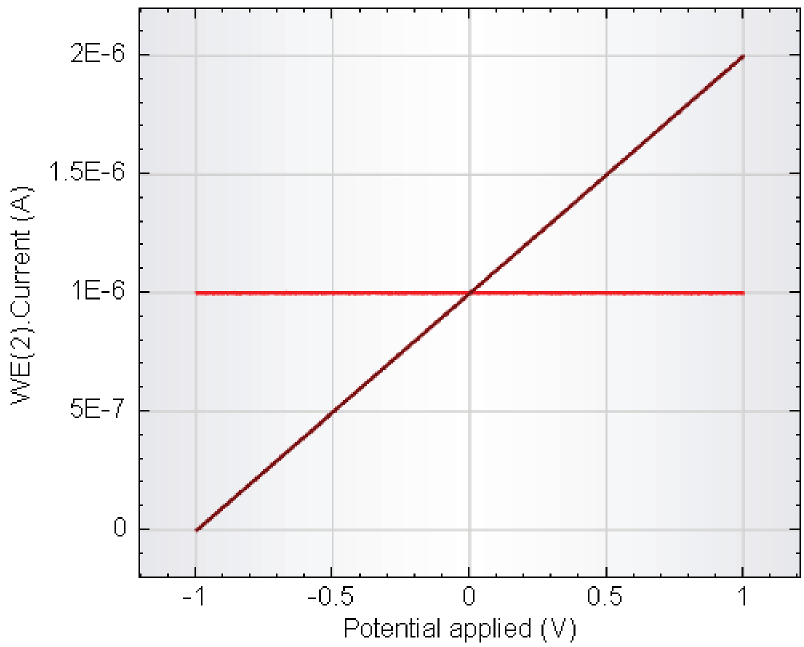


Figure 1.62 – The expected result of the TestBA procedure (red curve: WE(2).Current (Bipot mode), brown curve: WE(2).Current (Scanning Bipot mode))

The test is successful if the measured data can be compared to the reference data.

1.6.4 – Test of BIPOT

The TestBIPOT procedure can be used to test the correct functionality of the BIPOT module.

Load the **TestBIPOT** procedure, connect WE(1) to dummy cell (a) and WE(2) to dummy cell (b) as shown in Figure 1.60 and press the start button.

A message will be displayed when the measurement starts. The test uses the cyclic voltammetry staircase method and performs a single potential scan. During this scan the potential of the WE(2) is controlled with respect to the potential of the reference electrode, with a potential offset of 1 V.

At the end of the measurement, switch to the Analysis view and load the data for evaluation.

The data set includes two groups of data points (see Figure 1.63).

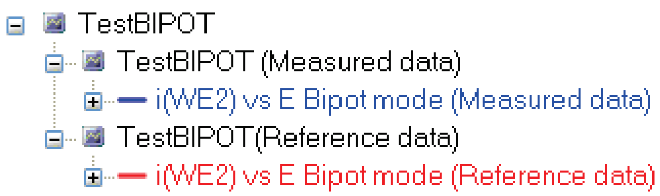


Figure 1.63 – The data obtained with the TestBIPOT procedure

The first group contains the measured data points. The other group contains data points for the WE(2).Current from a reference measurement. This data can be used for comparison with the data points obtained during the test.

The measured data should be similar to the reference data provided for comparison as shown in Figure 1.64.

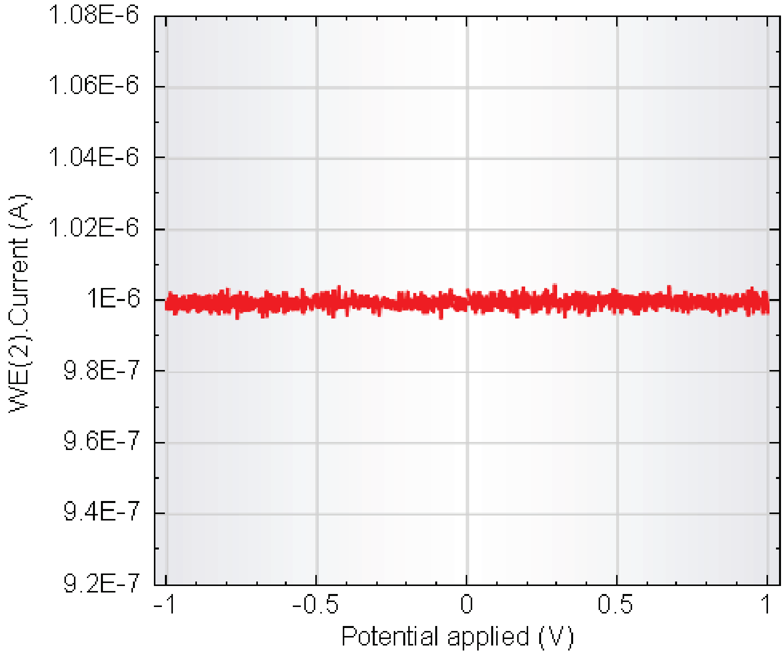


Figure 1.64 – The expected result of the TestBIPOT procedure

The test is successful if the measured data can be compared to the reference data.

1.6.5 – Test of ARRAY

The TestARRAY procedure can be used to test the correct functionality of the ARRAY module[17].

Load the **TestARRAY** procedure, connect WE(1) to dummy cell (a) and WE(2) to dummy cell (b) as shown in Figure 1.60 and press the start button.

A message will be displayed when the measurement starts. The test uses the cyclic voltammetry staircase method and performs a single potential scan. During this scan the potential of the WE(2) is controlled with respect to the potential of WE(1), with a potential offset of 1 V.

At the end of the measurement, switch to the Analysis view and load the data for evaluation.

[17] If the BIPOT module is equipped with a switch on the front panel of the instrument, the TestBIPOT can be used to test the bipotentiostat mode and the TestARRAY can be used to test the scanning bipotentiostat mode.

The data set includes two groups of data points (see Figure 1.65).

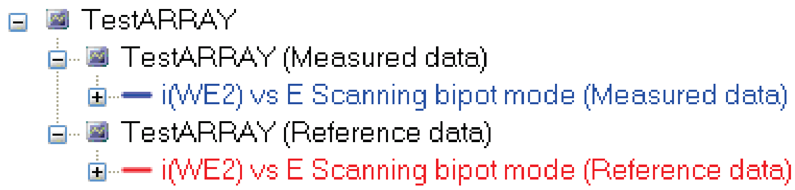


Figure 1.65 – The data obtained with the TestARRAY procedure

The first group contains the measured data points. The other group contains data points for the WE(2).Current from a reference measurement. This data can be used for comparison with the data points obtained during the test.

The measured data should be similar to the reference data provided for comparison as shown in Figure 1.66.

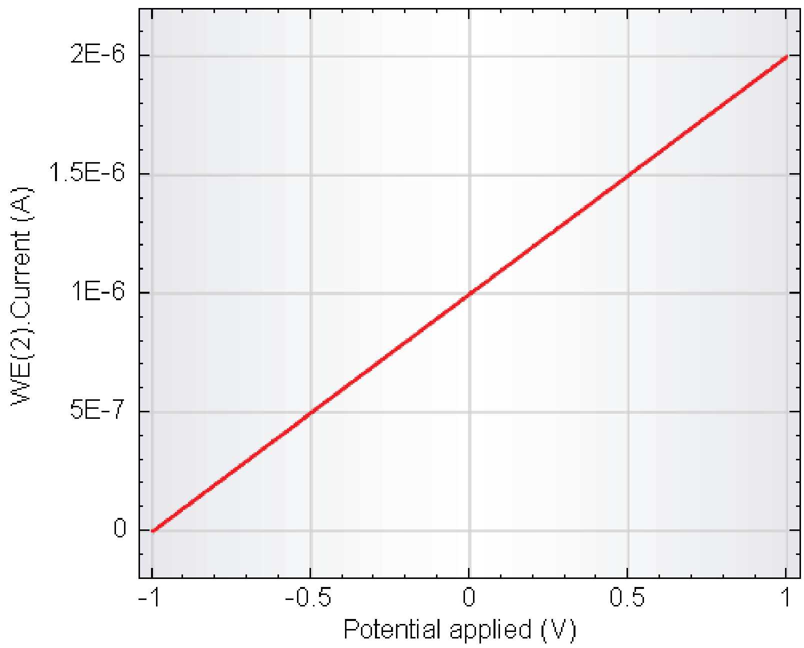


Figure 1.66 – The expected result of the TestARRAY procedure

The test is successful if the measured data can be compared to the reference data.

1.6.6 – Test of the Booster10A and the Booster20A

The TestBooster10A and TestBooster20A procedures can be used to test the correct functionality of the Booster10A and Booster20A, respectively. Before these tests can be performed, make sure that the hardware setup is defined properly and that the Booster is installed correctly.

Load the **TestBooster10A** or **TestBooster20A** procedure depending on the type of Booster. Connect the PGSTAT and the Booster to the special booster test cell. Press the start button to begin the measurement.

A message will be displayed when the measurement starts. The test uses the cyclic voltammetry staircase method and performs a single potential scan. During this scan the potential of the working electrode is scanning between -1 V and 1 V.

At the end of the measurement, switch to the Analysis view and load the data for evaluation.

The data set includes two groups of data points (see Figure 1.67).

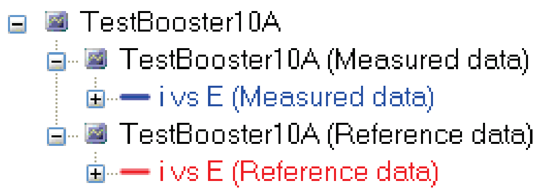


Figure 1.67 – The data obtained with the TestBooster10A procedure

Note

The data for the TestBooster20A is displayed in a similar way.

The first group contains the measured data points. The other group contains data points from a reference measurement. This data can be used for comparison with the data points obtained during the test.

The measured data should be similar to the reference data provided for comparison as shown in Figure 1.68.

Note

Small deviation can be observed between the measured data points and the reference data because of the tolerance of the resistance included in the special booster test cell (± 5%).

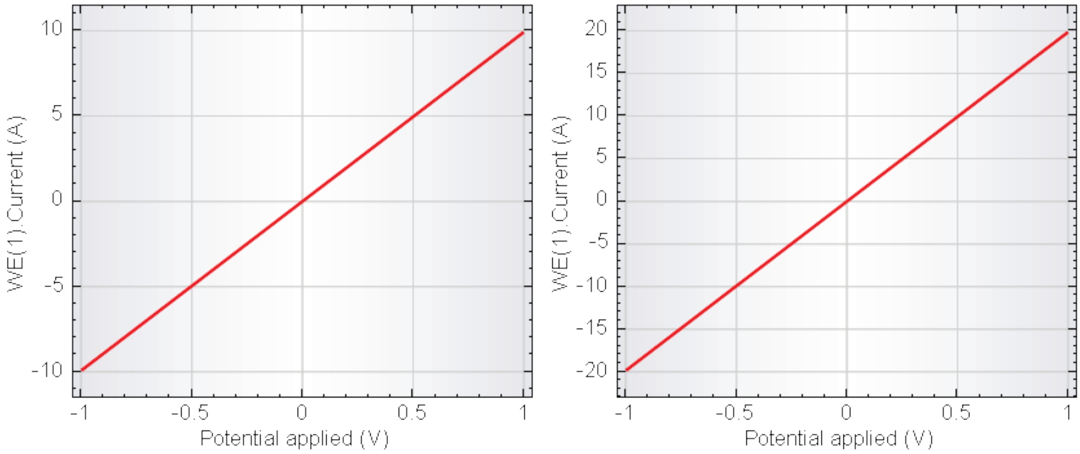


Figure 1.68 – The expected result of the TestBooster10A procedure (left) and the TestBooster20A procedure (right)

The test is successful if the measured data can be compared to the reference data.

1.6.7 – Test of ECD

The TestECD procedure can be used to test the correct functionality of the ECD module.

Load the **TestECD** procedure, connect WE(1) to dummy cell (a) and press the start button.

A message will be displayed when the measurement starts. The test uses the cyclic voltammetry staircase method and performs a single potential scan. During this scan the potential of the working electrode is scanning between -1 V and 1 V.

At the end of the measurement, switch to the Analysis view and load the data for evaluation.

The data set includes two groups of data points (see Figure 1.69).

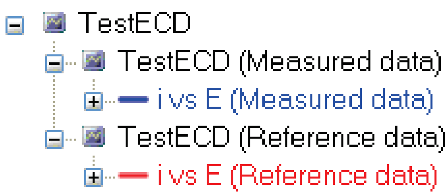


Figure 1.69 – The data obtained with the TestECD procedure

The first group contains the measured data points. The other group contains data points from a reference measurement. This data can be used for comparison with the data points obtained during the test.

The measured data should be similar to the reference data provided for comparison as shown in Figure 1.70.

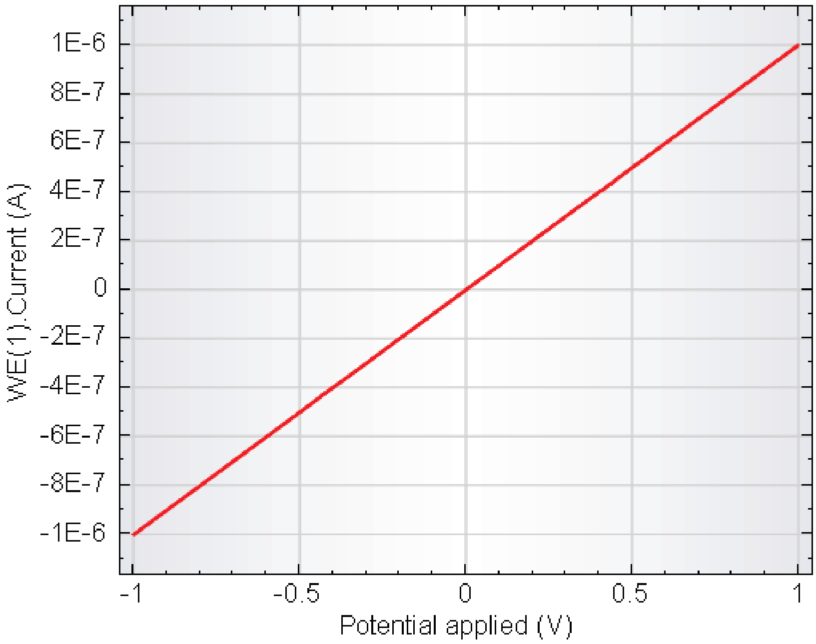


Figure 1.70 – The expected result of the TestECD procedure

The test is successful if the measured data can be compared to the reference data.

1.6.8 – Test of ECN

The TestECN procedure can be used to test the correct functionality of the ECN module.

Load the **TestECN** procedure, connect the ECN cable to the --> E input of the ECN module. Connect the red plug of the ECN cable to dummy cell (a). Connect the black plug of the ECN cable to the CE connector of the dummy cell. Connect the RE, CE and S and WE from the PGSTAT to dummy cell (a) (see Figure 1.71).

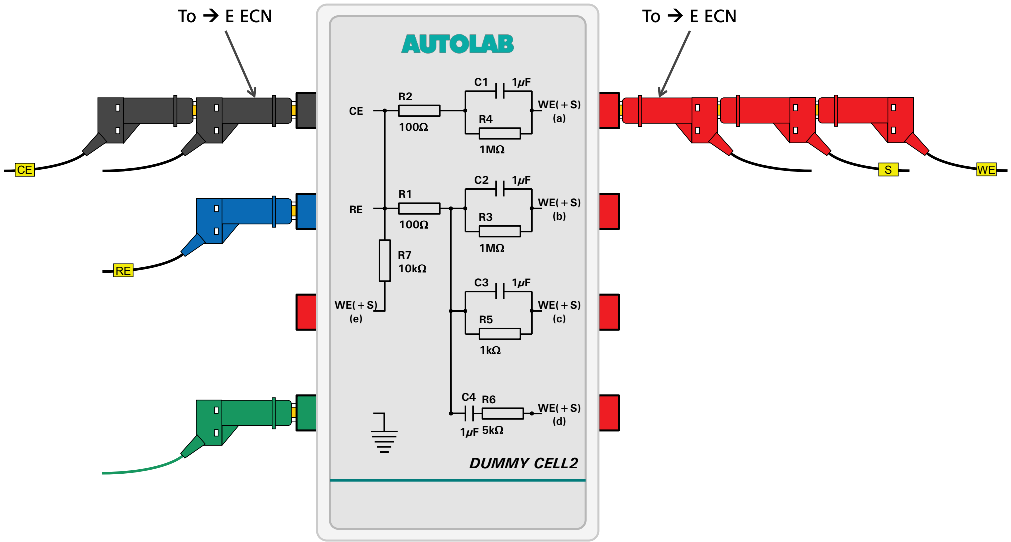


Figure 1.71 – Overview of the connections to the dummy cell required for the TestECN procedure

Press the start button to start the measurement. A message will be displayed when the measurement starts. The test uses the cyclic voltammetry staircase method and performs a single potential scan. During this scan the potential of the working electrode is scanning between -1 V and 1 V. The potential between the counter electrode and the working electrode is recorded by the ECN module.

At the end of the measurement, switch to the Analysis view and load the data for evaluation.

The data set includes two groups of data points (see Figure 1.72).

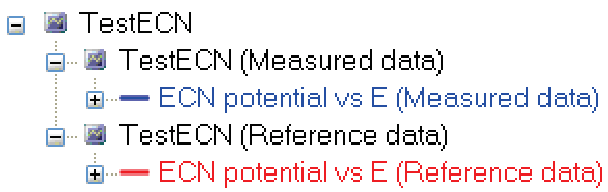


Figure 1.72 – The data obtained with the TestECN procedure

The first group contains the measured data points. The other group contains data points from a reference measurement. This data can be used for comparison with the data points obtained during the test.

The measured data should be similar to the reference data provided for comparison as shown in Figure 1.73.

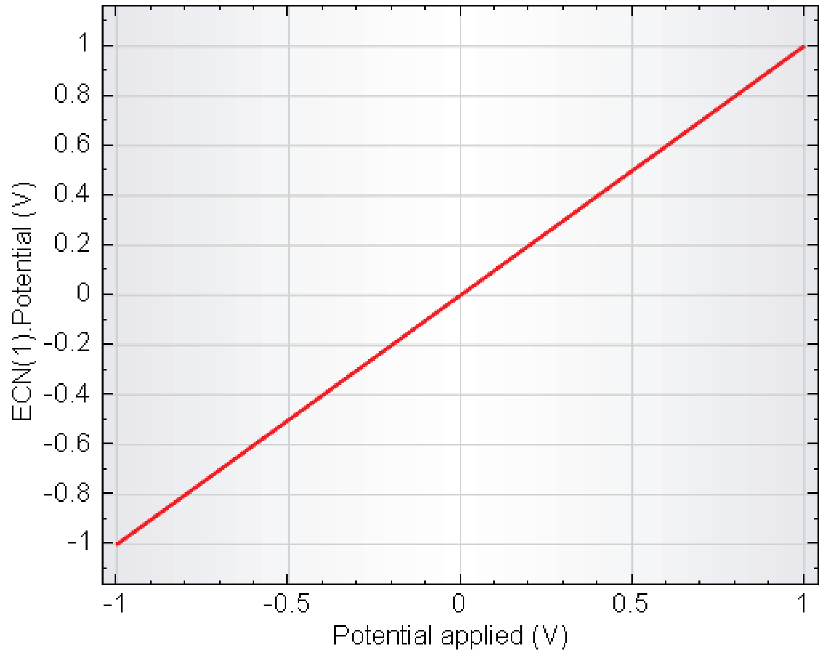


Figure 1.73 – The expected result of the TestECN procedure

The test is successful if the measured data can be compared to the reference data.

1.6.9 – Test of FI20-Filter

The TestFI20-Filter procedure can be used to test the correct functionality of the filter circuit of the FI20-Filter module.

Load the **TestFI20-Filter** procedure, connect dummy cell (a) and press the start button.

A message will be displayed when the measurement starts. The test uses the cyclic voltammetry staircase method and performs a single potential scan. During this scan the potential of the working electrode is scanning between -1 V and 1 V. During this measurement, the filter is switched on and a filter time-constant of 0.1 s is used.

At the end of the measurement, switch to the Analysis view and load the data for evaluation.

The data set includes two groups of data points (see Figure 1.74).

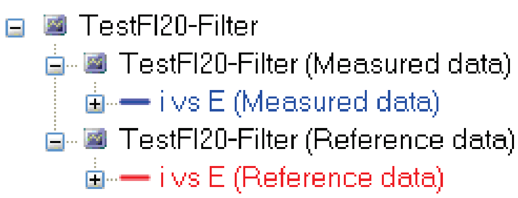


Figure 1.74 – The data obtained with the TestFI20-Filter procedure

The first group contains the measured data points. The other group contains data points from a reference measurement. This data can be used for comparison with the data points obtained during the test.

The measured data should be similar to the reference data provided for comparison as shown in Figure 1.75.

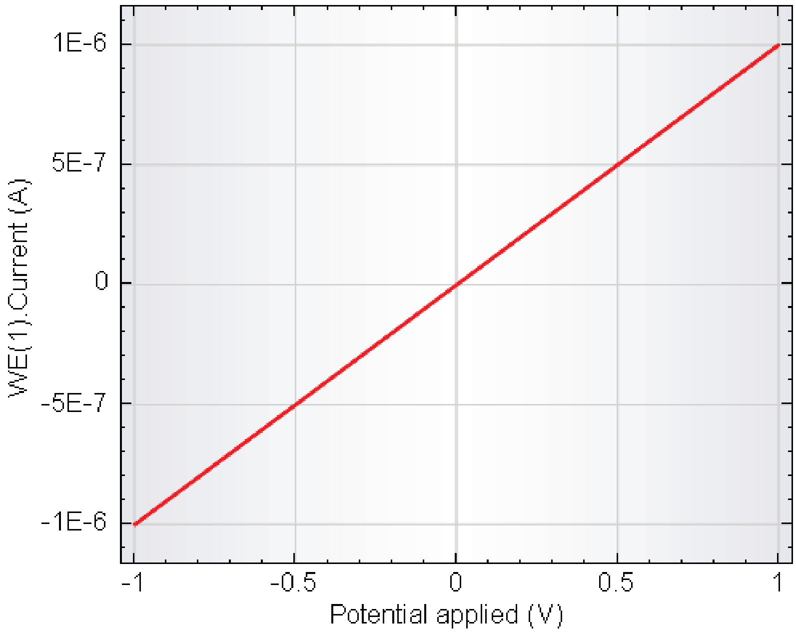


Figure 1.75 – The expected result of the TestFI20-Filter procedure

The test is successful if the measured data can be compared to the reference data.

1.6.10 – Test of FI20-Integrator

The TestFI20-Integrator procedure can be used to test the correct functionality of the integrator circuit of the FI20-Integrator module for the Autolab PGSTAT series (except the PGSTAT101 for which a specific test is provided, see Section 1.6.11) and the µAutolab II and III.

Note

The FI20-Integrator needs to be properly calibrated before the test. Integrator calibration is performed in the Diagnostics application. Please refer to Section 1.5 of the Getting Started manual or the FI20 tutorial for more information.

Load the **TestFI20-Integrator** procedure, connect dummy cell (a) and press the start button.

A message will be displayed when the measurement starts. The test uses the cyclic voltammetry current integration staircase method and performs a single potential scan. During this scan the potential of the working electrode is scanning between -1 V and 1 V. During this measurement, an integration time-constant of 0.01 s is used.

At the end of the measurement, switch to the Analysis view and load the data for evaluation.

The data set includes two groups of data points (see Figure 1.76).

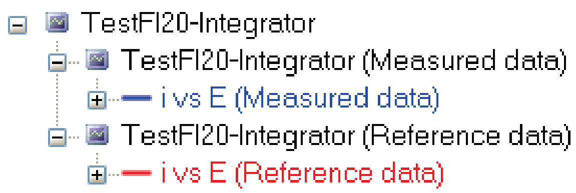


Figure 1.76 – The data obtained with the TestFI20-Integrator procedure

The first group contains the measured data points. The other group contains data points from a reference measurement. This data can be used for comparison with the data points obtained during the test.

The measured data should be similar to the reference data provided for comparison as shown in Figure 1.77.

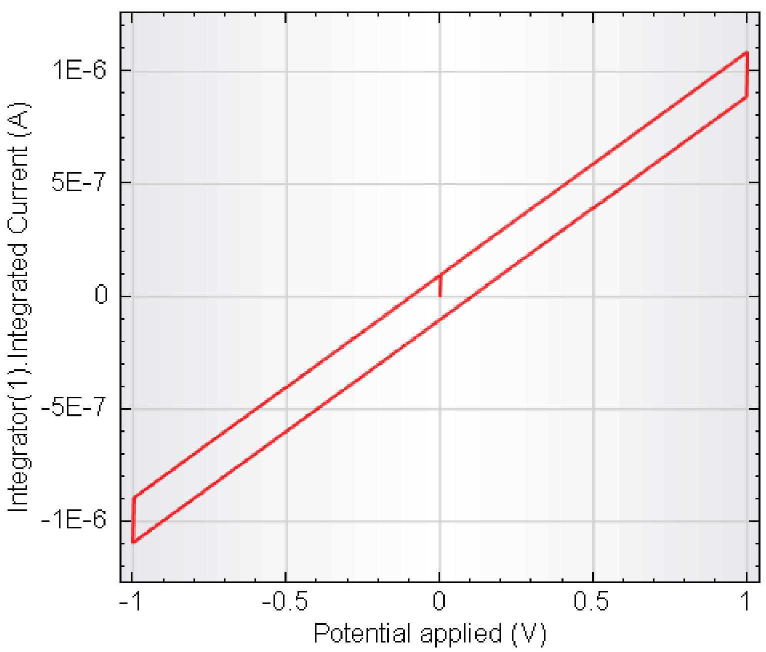


Figure 1.77 – The expected result of the TestFI20-Integrator procedure

The test is successful if the measured data can be compared to the reference data.

Note

The current recorded during current integration cyclic voltammetry strongly depends on the value of the capacitance included in the circuit of dummy cell (a). This capacitance has a tolerance of ± 5 %. The measured data points should therefore by qualitatively compared to the reference data provided with the test.

1.6.11 – Test of FI20-Integrator-PGSTAT101

The TestFI20-Integrator-PGSTAT101 procedure can be used to test the correct functionality of the on-board integrator of the PGSTAT101 and the Autolab M101 potentiostat/galvanostat module.

Note

The FI20-Integrator needs to be properly calibrated before the test. Integrator calibration is performed in the Diagnostics application. Please refer to Section 1.5 of the Getting Started manual or the FI20 tutorial for more information.

Warning

This test is designed for the PGSTAT101 and M101 only. For all the other Autolab instruments fitted with a FI20 module, please use the **TestFI20-Integrator** procedure (see Section 1.6.10).

Load the **TestFI20-Integrator-PGSTAT101** procedure.

This test uses the internal dummy cell of the instrument. Connect the CE and the RE electrode leads and the WE and S from the cell cable as shown in Figure 1.78 and press the start button.

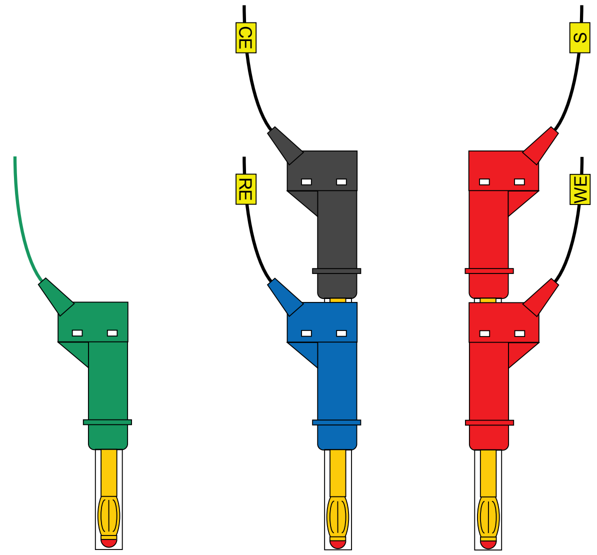


Figure 1.78 – The connections required for the TestFI20-Integrator-PGSTAT101 procedure

A warning message, indicating that the internal dummy cell is used, will be shown during validation (see Figure 1.79). This warning is provided as a reminder and the OK button can be clicked to proceed with the measurement.

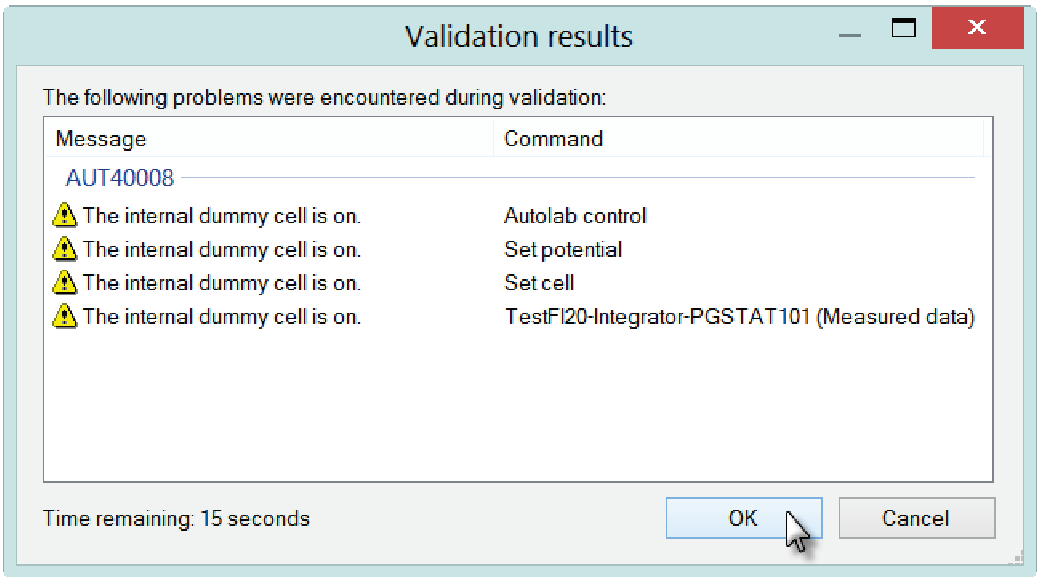


Figure 1.79 – A warning is displayed at the beginning of the procedure

A message will be displayed when the measurement starts (see Figure 1.80).

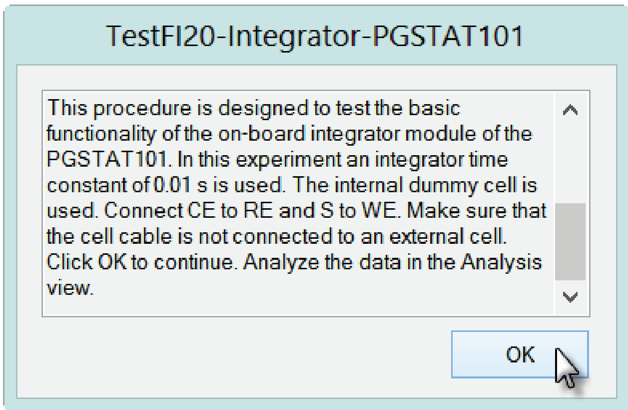


Figure 1.80 – A message is displayed at the beginning of the test

The test uses the cyclic voltammetry current integration staircase method and performs a single potential scan. During this scan the potential of the working electrode is scanning between -1 V and 1 V. During this measurement, an integration time-constant of 0.01 s is used.

At the end of the measurement, switch to the Analysis view and load the data for evaluation.

The data set includes two groups of data points (see Figure 1.81).

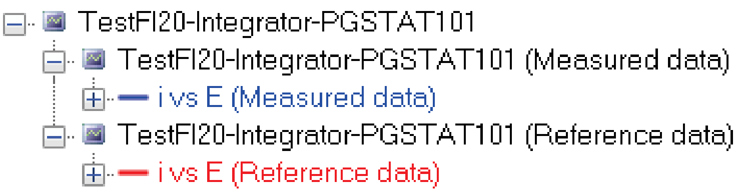


Figure 1.81 – The data obtained with the TestFI20-Integrator-PGSTAT101 procedure

The first group contains the measured data points. The other group contains data points from a reference measurement. This data can be used for comparison with the data points obtained during the test.

The measured data should be similar to the reference data provided for comparison as shown in Figure 1.82.

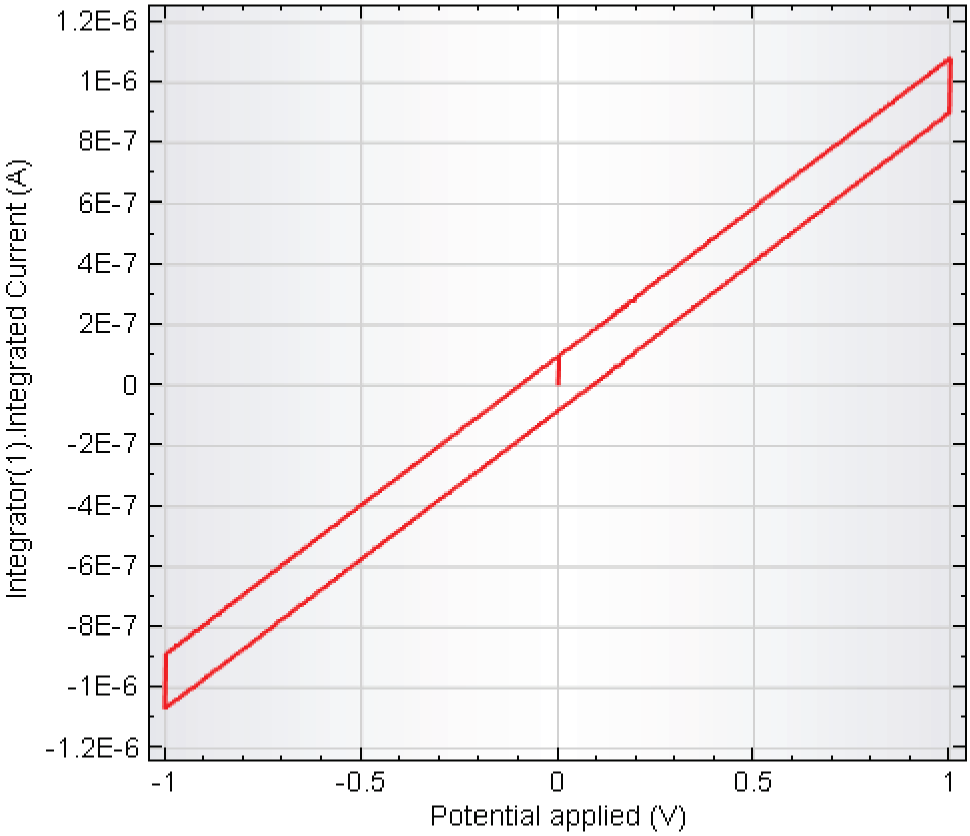


Figure 1.82 – The expected result of the TestFI20-Integrator-PGSTAT101 procedure

The test is successful if the measured data can be compared to the reference data.

Note

The current recorded during current integration cyclic voltammetry strongly depends on the value of the capacitance included in the circuit of dummy cell (a). This capacitance has a tolerance of ± 5 %. The measured data points should therefore be qualitatively compared to the reference data provided with the test.

1.6.12 – Test of FRA

The TestFRA procedure can be used to test the correct functionality of the FRA32M and the FRA2 module[18].

[18] When the FRA32M or FRA2 is installed in a PGSTAT302F, make sure that the PGSTAT302F is set to Normal mode.

Warning

For the FRA2 module make sure that the **FRA2 offset DAC range** property is set properly in the hardware setup. For FRA2 modules, the correct value is 5 V. For FRA2.V10 modules, the correct value is 10 V. Failure to set this value properly may result in faulty data at frequencies of 25 Hz and lower (refer to front panel labels of the FRA2 module on the instrument).

Load the **TestFRA** procedure, connect dummy cell (c) and press the start button.

A message will be displayed when the measurement starts. The test uses a potentiostatic frequency scan from 10 kHz to 0.1 Hz with a 10 mV amplitude. The frequency scan contains 50 frequencies with a logarithmic distribution. The measurement takes about four minutes to finish.

Click the OK button to continue with the measurement. During the experiment, four plots are shown in the measurement view (see Figure 1.83). Plot #1 corresponds to the Nyquist plot (-Z'' vs Z'), plot #2 corresponds to the Bode plot (|Z| and -ϕ vs frequency), plot #3 corresponds to the resolution plot (i(resolution) vs t and E(resolution) vs t) and plot #4 corresponds to the Lissajous plot (i(AC) vs E(AC)).

Note

Switch the measurement view to *Four plots* mode by pressing the button in the toolbar.

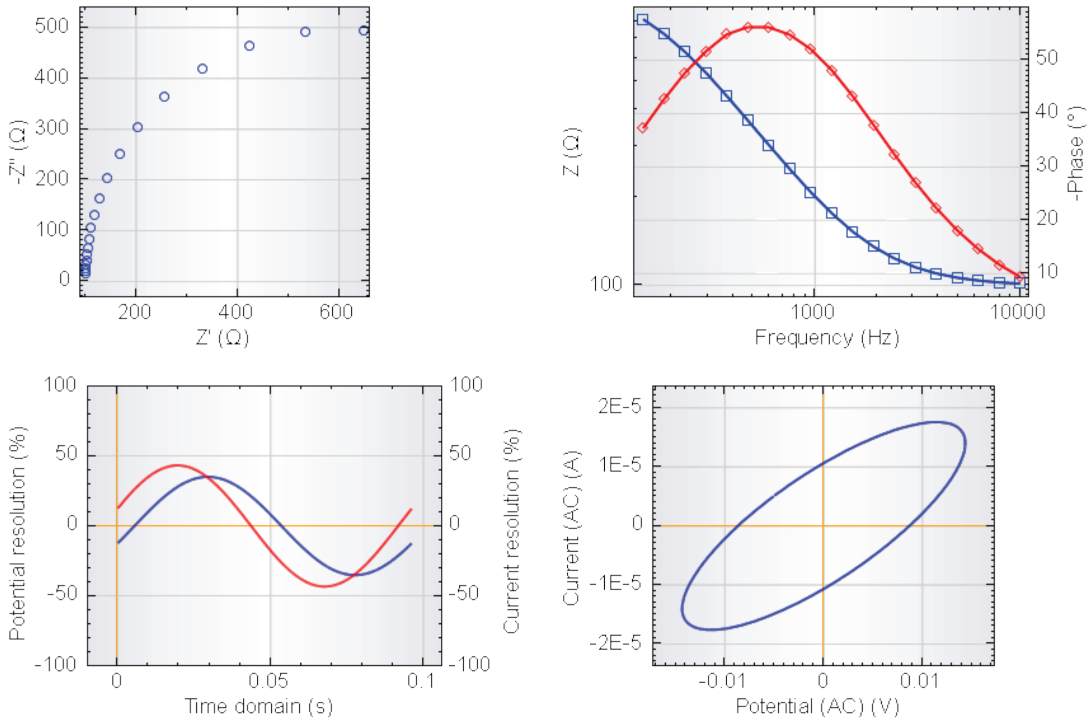


Figure 1.83 – The measured values are displayed as a Nyquist plot (plot #1), Bode plot (plot #2), Resolution plot vs time (plot #3) and Lissajous plot (plot #4)

At the end of the measurement, the data is automatically fitted using a R(RC) equivalent circuit and the calculated values of the circuit elements are displayed in a message box (see Figure 1.84).

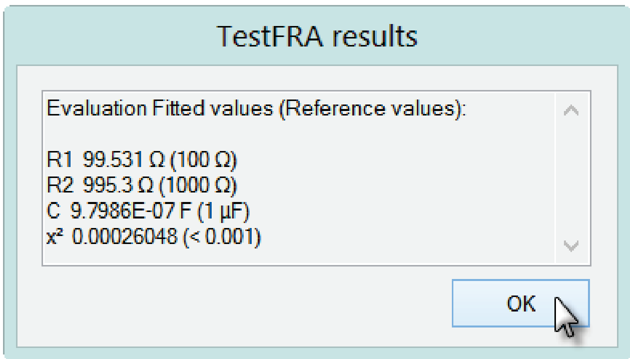


Figure 1.84 – The fitted values are shown in a message box at the end of the measurement (the reference values are shown in round brackets)

Reference values are shown in round brackets in the message box. The resistance values should be within ± 1% of the reference value and the capacitance value should be within ± 5% of the reference value. The calculated χ^2 value should be smaller than 0.001.

Switch to the Analysis view to inspect the measured and fitted data in detail. The data set includes the measured data points and the result of an automatic fit of the impedance data with the R(RC) equivalent circuit (see Figure 1.85).

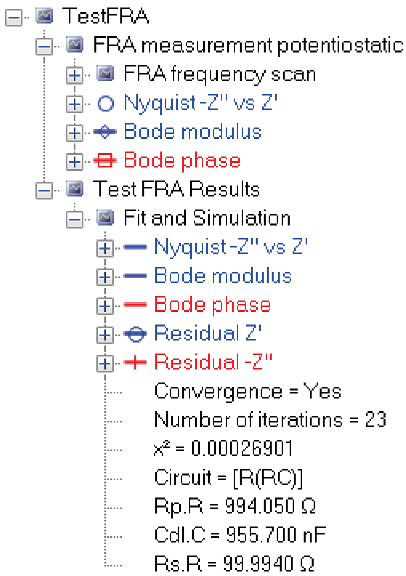


Figure 1.85 – The data obtained with the TestFRA procedure

The value of Rs, Rp, Cdl and χ^2 displayed in the explorer frame. Select the Fit and Simulation item in the data explorer and click the Edit button located on the left hand side of the plot area to open the Equivalent Circuit Editor window (see Figure 1.86).

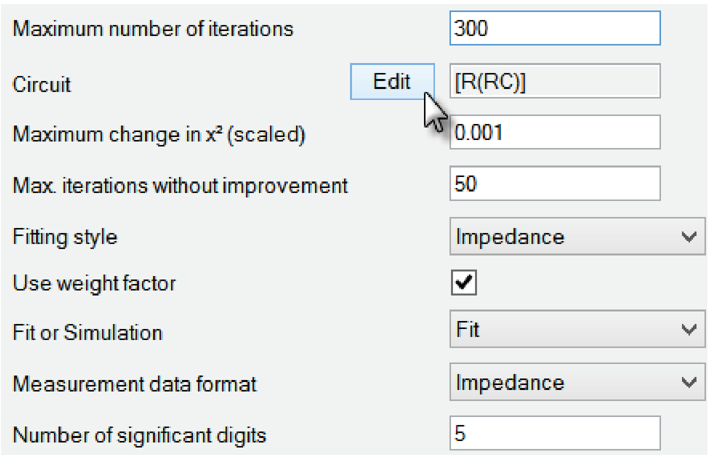


Figure 1.86 – Opening the results of the Fitting of the data

The results of the calculation are graphically shown in the Equivalent Circuit Editor. Select the Generate Report option from the Tools menu to display a short report table for the fitted data (see Figure 1.87). The values shown in the last column corresponds to the estimated errors on the different circuit elements, in %.

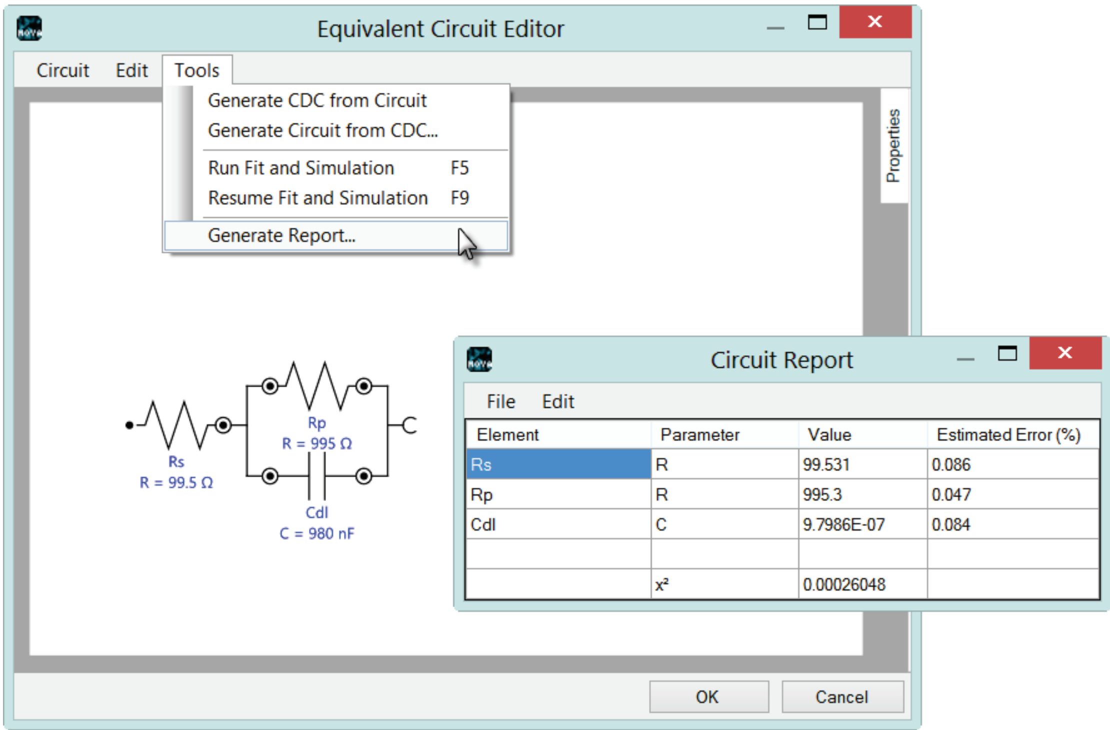


Figure 1.87 – The Equivalent Circuit Editor window can be used to display the details of the calculation

The errors on the estimated parameters from the fitting algorithm must be smaller than **0.2 %.**

1.6.13 – Test of MUX

The TestMUX procedure can be used to test the correct functionality of the MUX module. This procedure can be used to test any type of MUX configuration.

Load the **TestMUX** procedure, connect Channel 1 to dummy cell (a) and Channel 2 to dummy cell (c) as shown in Figure 1.88.

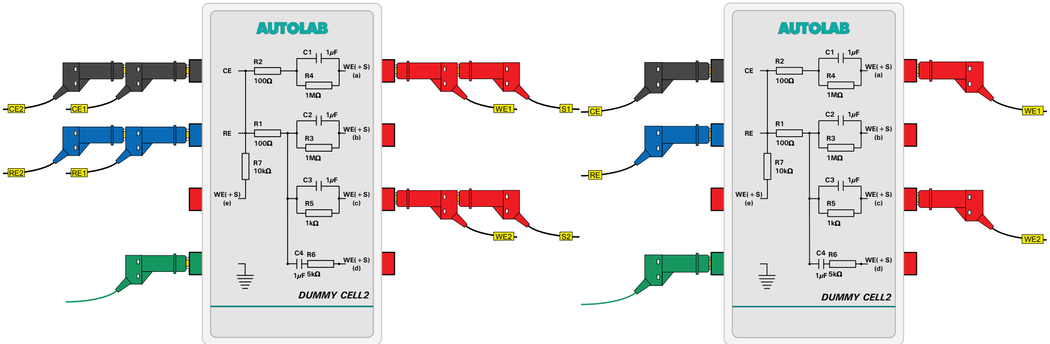


Figure 1.88 – Overview of the connections to the dummy cell required for the TestMUX procedure (left: MULTI4, right: SCNR16)

Press the start button. A message will be displayed when the measurement starts. The test uses the cyclic voltammetry staircase method and performs two single potential scans. The first scan is performed on Channel 1 and the second scan is performed on Channel 2.

During each scan the potential of the working electrode is scanning between -1 V and 1 V. The recorded data points for Channel 1 are displayed on plot #1 and the data points for Channel 2 are displayed on plot #2.

Note

Switch the measurement view to *Two plots vertically tiled* mode by pressing the button in the toolbar.

At the end of the measurement, switch to the Analysis view and load the data for evaluation.

The data set includes four groups of data points (see Figure 1.89).

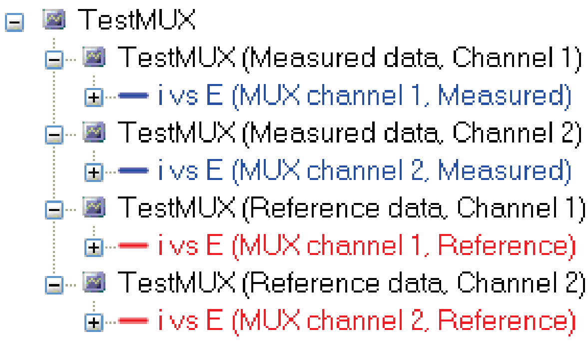


Figure 1.89 – The data obtained with the TestMUX procedure

The first two groups contain the measured on Channel #1 and on Channel #2. The other two groups contain data points for the WE(1).Current from a reference measurement. This data can be used for comparison with the data points obtained during the test.

The measured data should be similar to the reference data provided for comparison as shown in Figure 1.90.

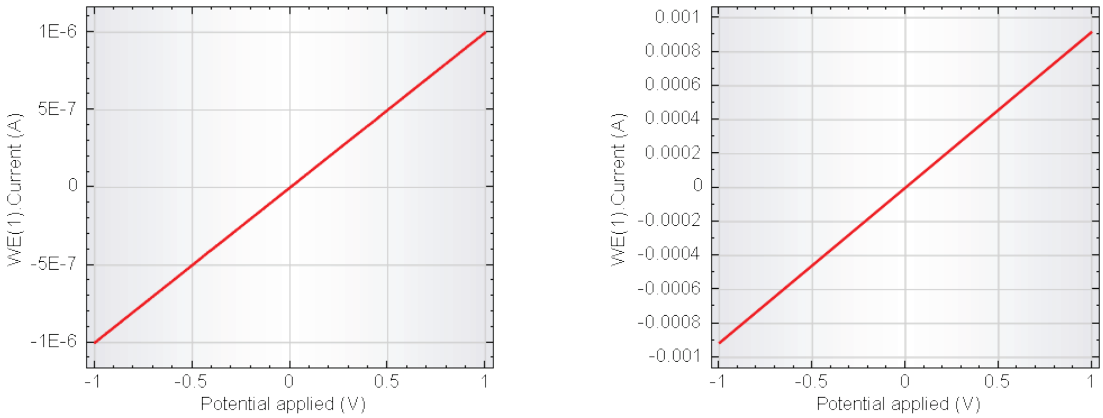


Figure 1.90 – The expected result of the TestMUX procedure (Channel 1 (left) and Channel 2 (right))

The test is successful if the measured data can be compared to the reference data.

1.6.14 – Test of pX and pX1000

The TestpX and TestpX1000 procedures can be used to test the correct functionality of the pX and pX1000 modules, respectively. Both tests are performed on the dummy cell.

Load the **TestpX** or the **TestpX1000** procedure depending on the module to test from the Standards database. Connect the pX/pX1000 cable to the module, on the front panel of the instrument. Connect the V+ lead from the pX/pX1000 cable (red lead) to dummy cell (a) and the V- lead from the pX/pX1000 cable (black lead) to the CE connector on the dummy cell. Connect the PGSTAT cables to dummy cell (a) (see Figure 1.91).

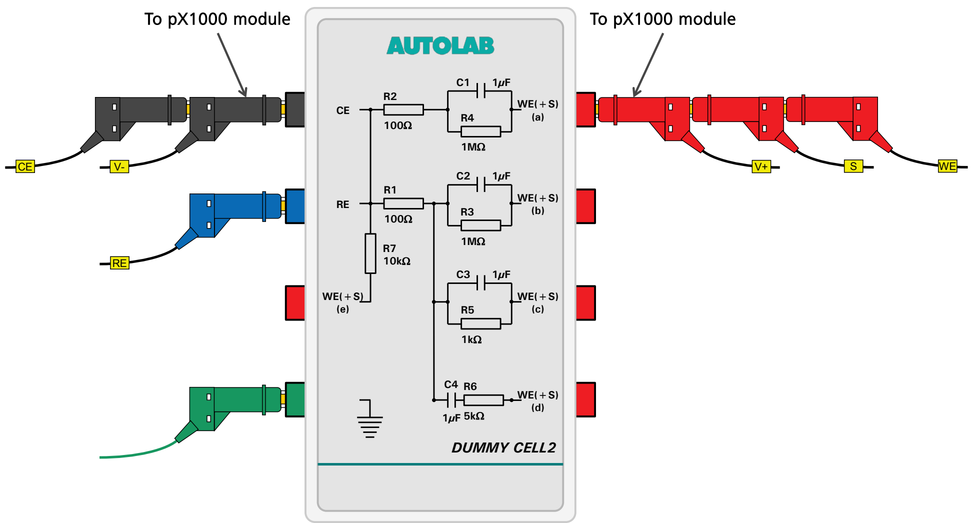


Figure 1.91 – Overview of the connections to the dummy cell required for the TestpX and the TestpX1000

Warning

During the **TestpX** procedure, designed to verify the functionality of the **pX** module, make sure that the 50 Ohm resistor BNC shunt is **NOT** connected to the ⊙ G BNC input on the front panel of the pX module.

Press the start button to start the measurement.

A message will be displayed when the measurement starts. The test uses the cyclic voltammetry staircase method and performs a single potential scan. During this scan the potential of the working electrode is scanning between -1 V and 1 V. The potential between the counter electrode and the working electrode is recorded by the pX/pX1000 module.

At the end of the measurement, switch to the Analysis view and load the data for evaluation.

The data set includes two groups of data points (see Figure 1.92).

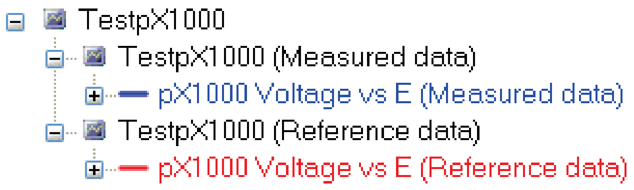


Figure 1.92 – The data obtained with the TestpX/TestpX1000 procedure

Note

The data for the TestpX is displayed in a similar way.

The first group contains the measured data points. The other group contains data points from a reference measurement. This data can be used for comparison with the data points obtained during the test.

The measured data should be similar to the reference data provided for comparison as shown in Figure 1.93.

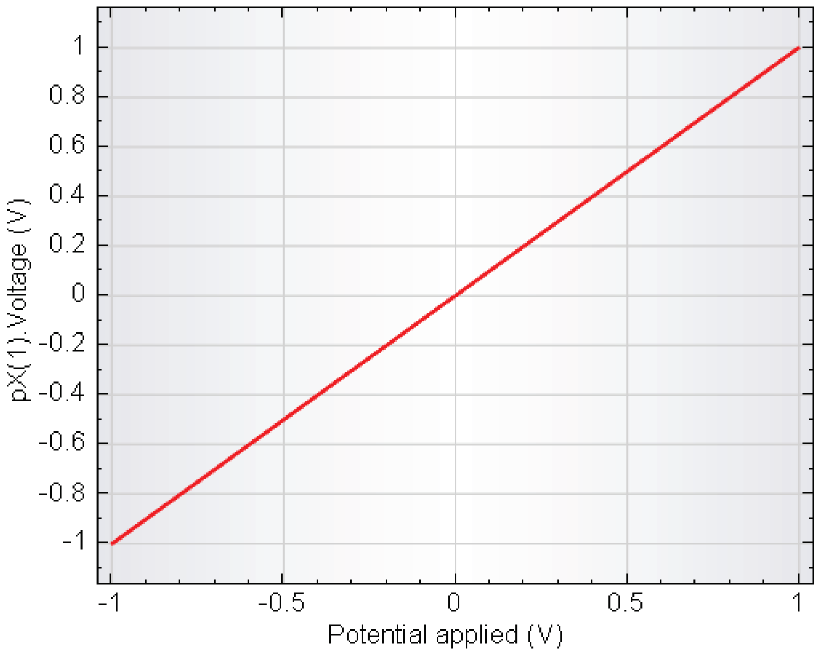


Figure 1.93 – The expected result of the TestpX or the TestpX1000 procedure

The test is successful if the measured data can be compared to the reference data.

1.6.15 – Test of the SCANGEN or the SCAN250

Two procedures, TestSCANGEN and TestSCAN250 can be used to test the correct functionality of the linear scan generator module (SCANGEN or SCAN250, respectively).

Load the **TestSCANGEN** or the **TestSCAN250** procedure depending on the module to test from the Standards database, connect dummy cell (a) and press the start button.

A message will be displayed when the measurement starts.

The test uses the cyclic voltammetry linear scan method and performs a potential scan starting from 0 V, between an upper vertex potential of 1 V and a lower vertex potential of -1 V. After the first potential scan, the measurement stops at the upper vertex potential, 1 V. At the end of the measurement, switch to the Analysis view and load the data for evaluation.

The data set includes two groups of data points (see Figure 1.94).

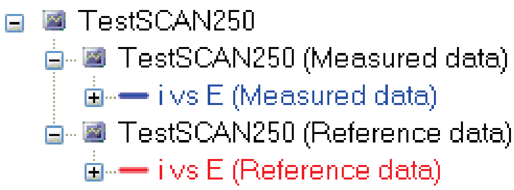


Figure 1.94 – The data obtained with the TestSCAN250 procedure

> **Note**
>
> The data for the TestSCANGEN is displayed in a similar way.

The first group, located under TestSCAN250 (Measured data) contains the measured current plotted versus the measured potential. The second group contains data from a reference measurement. This data can be used for comparison with the data points obtained during the test.

The measured data should be similar to the reference data provided for comparison as shown in Figure 1.95.

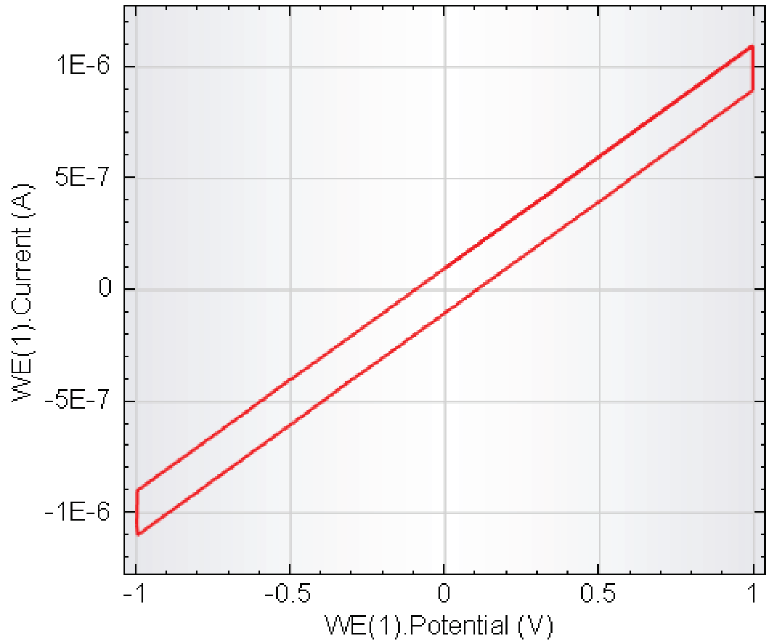


Figure 1.95 - The expected result of the TestSCAN250 or the TestSCANGEN procedure

The test is successful if the measured data can be compared to the reference data.

Note

the current recorded during a measurement with the SCANGEN or the SCAN250 module strongly depends on the value of the capacitance included in the circuit of dummy cell (a). This capacitance has a tolerance of ± 5 %. The measured data points should therefore be qualitatively compared to the reference data provided with the test.

1.6.16 – Test of the SCANGEN or the SCAN250 in combination with the ACD750 or the ADC10M

The TestADC/SCAN procedure can be used to test the correct functionality of the linear scan generator module (SCANGEN or SCAN250) in combination with the fast sampling ADC module (ADC750 or ADC10M) for high speed linear scan cyclic voltammetry measurements.

Load the **TestADC/SCAN** procedure, connect dummy cell (a) and press the start button.

A message will be displayed when the measurement starts.

Note

No data points can be shown real time during measurements with the fast-sampling ADC module.

The test uses the cyclic voltammetry linear scan high speed method and performs a potential scan starting from 0 V, between an upper vertex potential of 1 V and a lower vertex potential of -1 V. After the first potential scan, the measurement stops at the upper vertex potential, 1 V at 100 V/s. At the end of the measurement, switch to the Analysis view and load the data for evaluation.

The data set includes two groups of data points (see Figure 1.96).

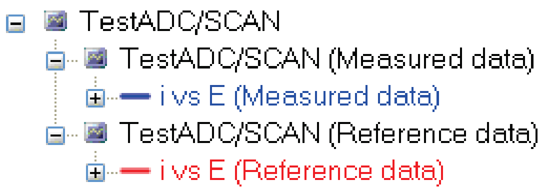


Figure 1.96 – The data obtained with the TestADC/SCAN procedure

The first group, located under TestADC/SCAN (Measured data) contains the measured current plotted versus the measured potential. The second group contains data from a reference measurement. This data can be used for comparison with the data points obtained during the test.

The measured data should be similar to the reference data provided for comparison as shown in Figure 1.97.

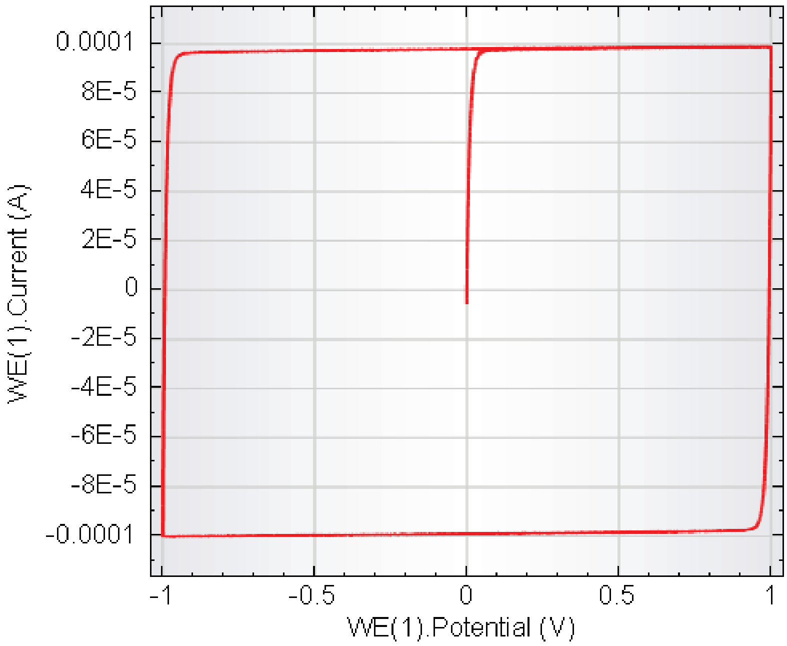


Figure 1.97 – The expected result of the TestADC/SCAN procedure

The test is successful if the measured data can be compared to the reference data.

Note

The current recorded during a measurement during the TestADC/SCAN procedure strongly depends on the value of the capacitance included in the circuit of dummy cell (a). This capacitance has a tolerance of ± 5 %. The measured data points should therefore be qualitatively compared to the reference data provided with the test.

1.6.17 – Test of the EQCM

The TestEQCM procedure can be used to test the correct functionality of the filter circuit of the EQCM.

Warning

This procedure cannot be performed on the dummy cell and it requires about 2 ml of water.

Load the **TestEQCM** procedure and insert a 6 MHz EQCM crystal in the EQCM cell. Fill the cell with ca. 2 ml of water and check for leakage. Connect the cell to the EQCM oscillator and the oscillator to the Autolab PGSTAT using the provided cable. Leave the cell connectors from the PGSTAT disconnected. Please refer to the EQCM user manual for more information.

Press the start button to start the measurement. Two messages will be displayed when the measurement starts (see Figure 1.98).

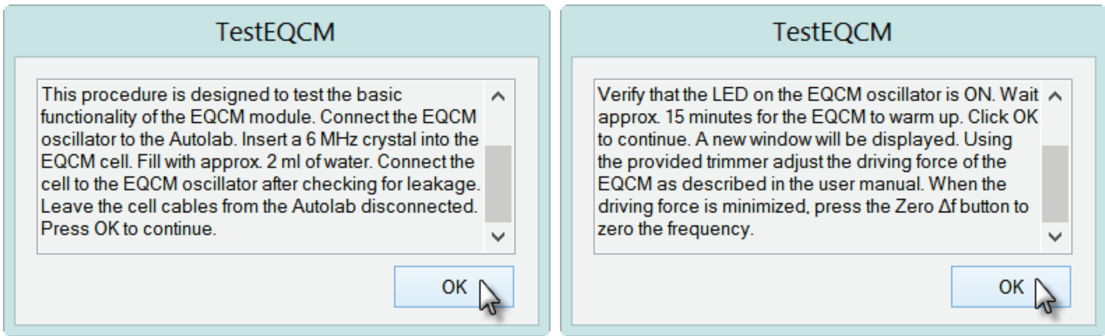


Figure 1.98 – Two messages are displayed at the beginning of the measurement

When the second message appears, verify that the LED on the EQCM oscillator box is **ON** (red or green).

Warning

Wait 15 minutes for the EQCM to warm up.

Click OK to continue. The *Determine EQCM zero frequency* window will appear (see Figure 1.99).

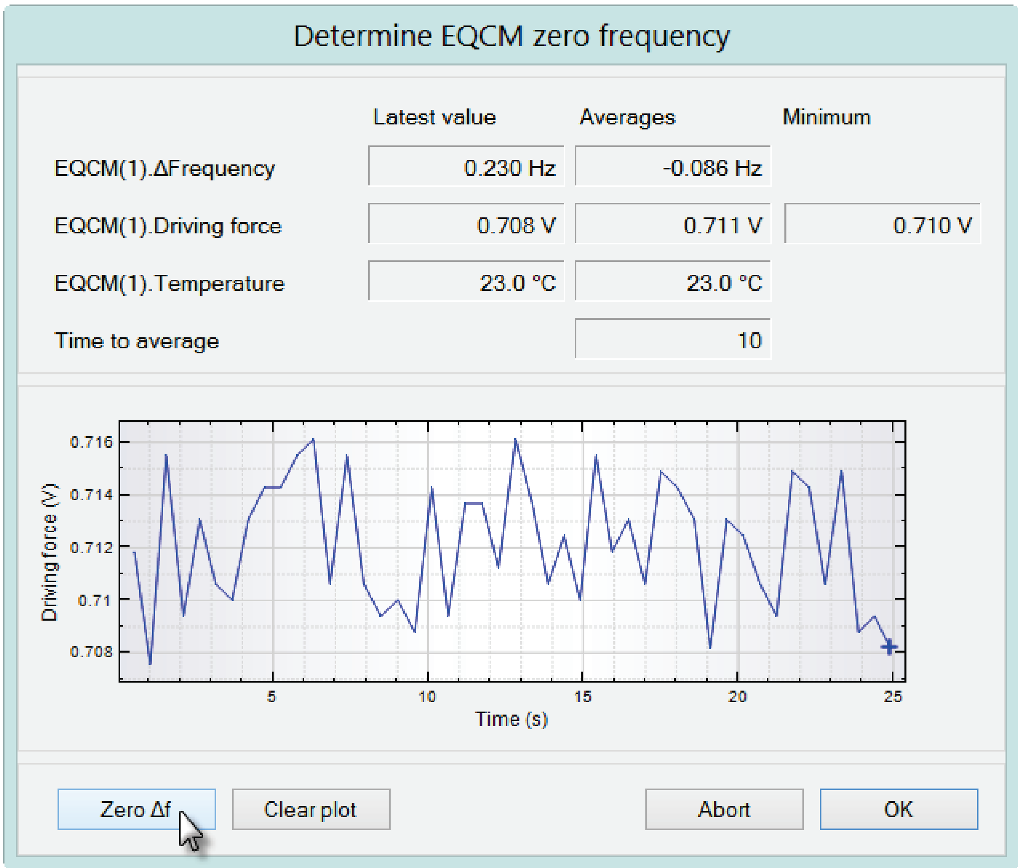


Figure 1.99 – The determine EQCM zero frequency window can be used to adjust the driving force

Using the provided adjustment tool, rotate the trimmer on the EQCM oscillator in order to minimize the driving force (refer to the EQCM user manual for more information).

When the driving force has been properly minimized, the LED on the EQCM oscillator must be green. Click the Zero Δf button in the *Determine EQCM zero frequency* window to zero the value of the EQCM(1).ΔFrequency signal.

After minimizing the ΔFrequency signal click the OK button to proceed with the measurement. The procedure records the three signals provided by the EQCM module during ten seconds. The EQCM(1).ΔFrequency and EQCM(1).Temperature signals are shown on plot #1 and the EQCM(1).Driving force signal is shown on plot #2 (see Figure 1.100).

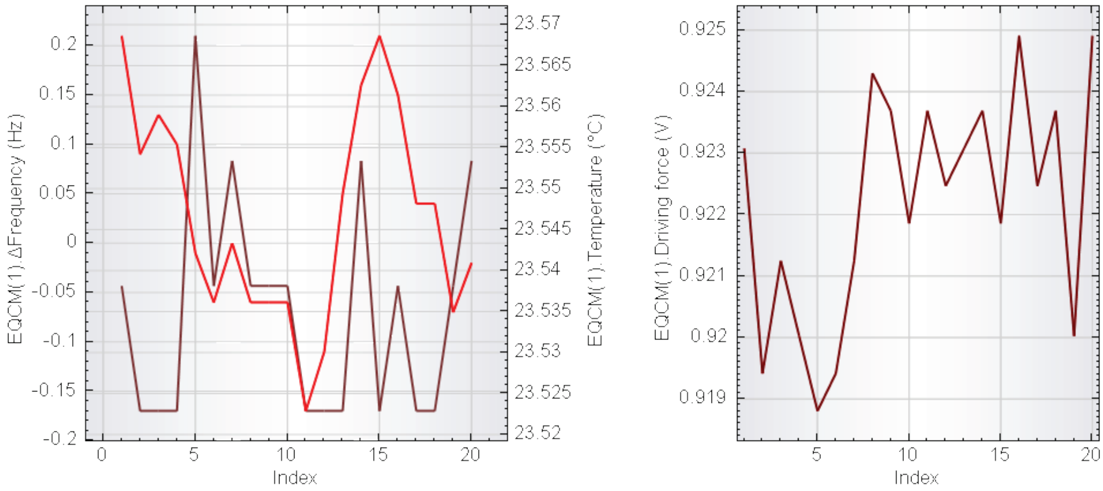


Figure 1.100 – The data recorded during the TestEQCM procedure

Note

Switch the measurement view to *Two plots vertically tiled* mode by pressing the button in the toolbar.

At the end of the measurement, a message is displayed, providing qualitative validation criteria for the measured data (see Figure 1.101).

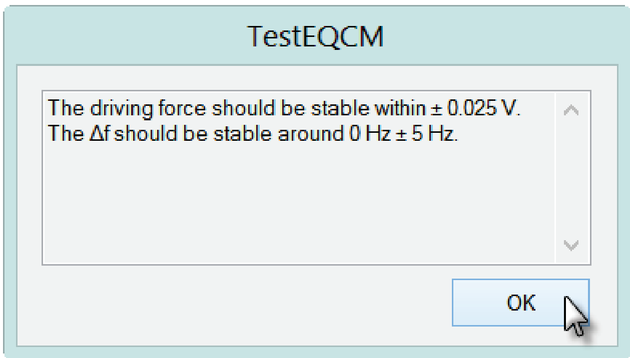


Figure 1.101 – A message is displayed at the end of the measurement

Switch to the Analysis view and load the data for evaluation. The data set includes two groups of data points (see Figure 1.102).

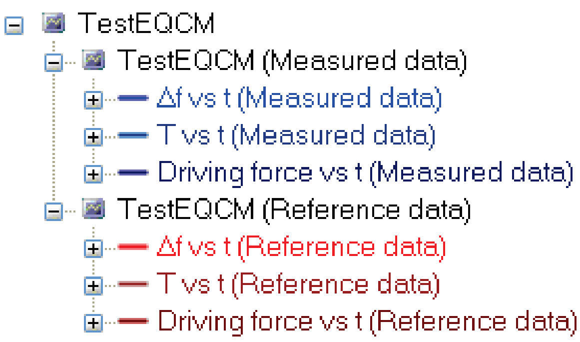


Figure 1.102 – The data obtained with the TestEQCM procedure

The first group contains the measured data points. The other group contains data points from a reference measurement. This data can be used for comparison with the data points obtained during the test.

The EQCM measurements depend on the temperature and the crystal used during the experiment. Comparison with the provided reference data points should be performed qualitatively.

1.6.18 – Determination of the C1 and C2 factors of the Autolab

When the FRA32M or FRA2 module is used in combination with the Autolab[19], the C1 and C2 factors need to be determined. These factors can be determined with the following procedures, included in the Module test database:

- PGSTAT C1 calibration
- PGSTAT C2 calibration

These procedures can be used in combination with the Autolab dummy cell.

Note

The C1 and C2 are predetermined when FRA32M or FRA2 module is preinstalled. The C1 and C2 factors must be determined experimentally when a FRA32M or FRA2 module is installed into an existing instrument. This determination must only be carried out upon installation of the module.

Note

The C1 and C2 determination in not required for the M101 and the PGSTAT204.

[19] When the FRA32M or FRA2 is installed in a PGSTAT302F, make sure that the PGSTAT302F is set to Normal mode.

The typical values of C1 and C2 are listed in Table 1.3 for the different Autolab instruments.

Instrument type	C1	C2	
PGSTAT302N	1.6E-11	3.0E-13	
PGSTAT302F	1.6E-11	1.0E-12	
PGSTAT128N	2.6E-11	1.0E-12	
PGSTAT128N	1.6E-11	1.0E-12	For instruments with serial number > AUT84179
PGSTAT100N	1.6E-11	5.0E-13	

Table 1.3 – Typical values for C1 and C2

Note

The determination of the C1 and C2 values is not required for the M101 module used in combination with the FRA32M module in the Multi Autolab instrument.

Before starting the determination of C1 and C2, verify that the starting values are set to 0. Open the hardware setup (Tools – Hardware setup), select instrument type in the **Main module** frame in the hardware setup window and adjust the value of C1 and C2 to 0, as shown in Figure 1.103.

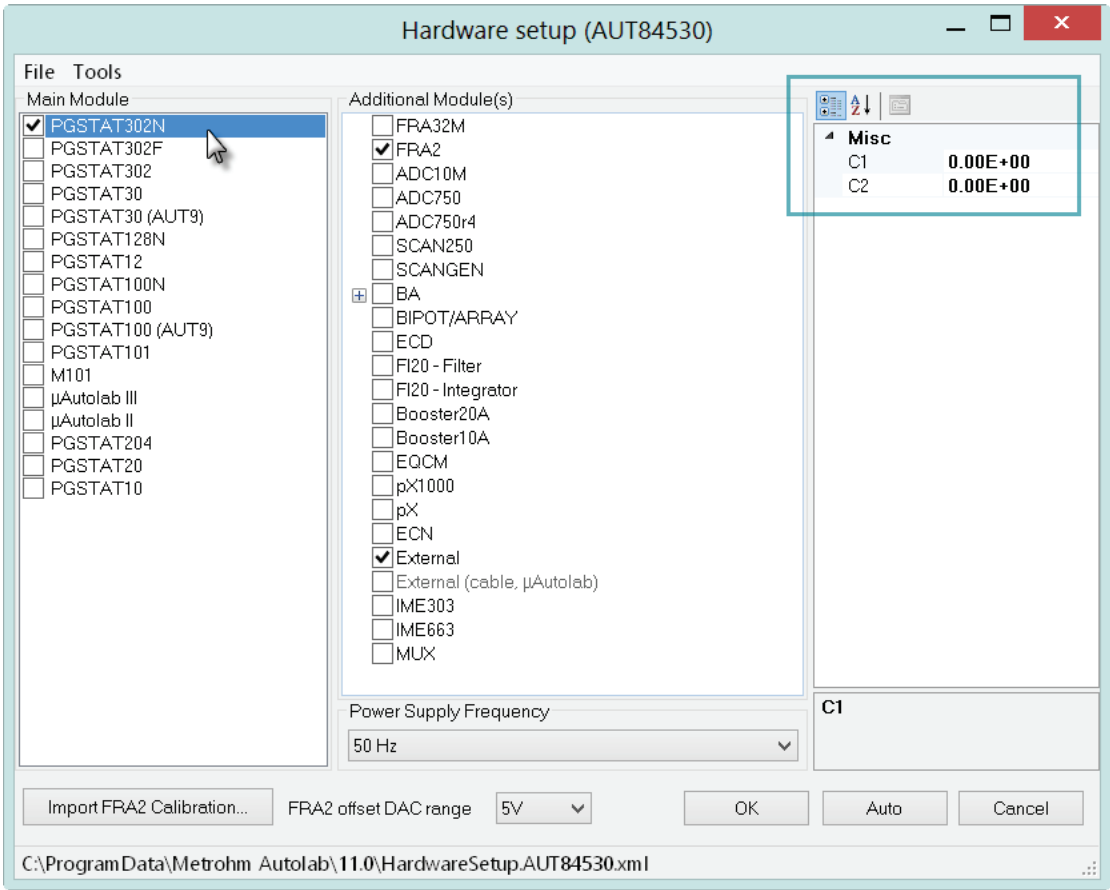


Figure 1.103 – The value of C1 and C2 must be set to 0 before starting the measurements

The determination of C1 and C2 requires the following items:

- Autolab Dummy cell
- Faraday cage[20]

1.6.18.1 – Determination of C1

Follow the steps described in this section to determine the value of the **C1** parameter.

1. Start the Nova software.
2. Load the procedure *PGSTAT C1 calibration* from the *Module test* database.
3. Connect the Autolab Dummy cell as shown in Figure 1.104. Connect the ground lead from the PGSTAT to the **Faraday cage**.

Note

Do not connect the ground lead from the PGSTAT to the Dummy cell. Place the dummy cell in the Faraday cage.

[20] Please contact your Autolab distributor if you need assistance.

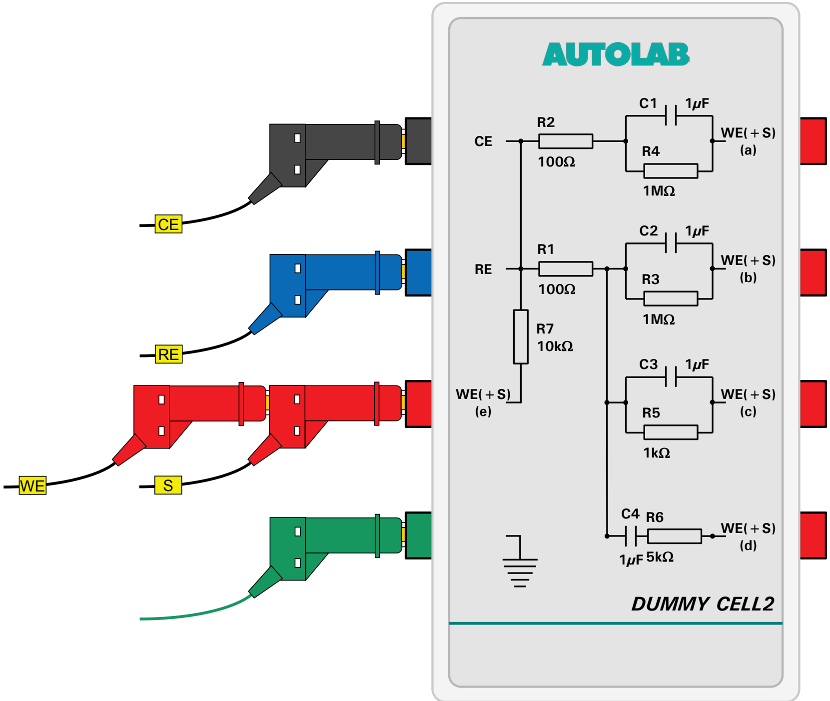


Figure 1.104 – Overview of the connections required for the determination of C1

4. Start the measurement and wait until it has been finished. Ignore the warning displayed during the procedure validation (see Figure 1.105).

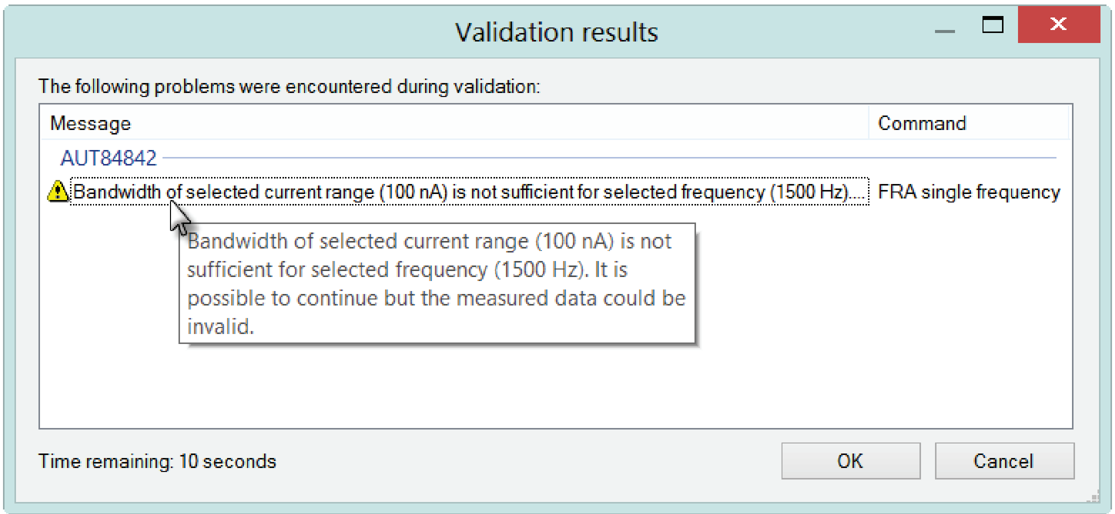


Figure 1.105 – Ignore the warning shown during the validation of the procedure

5. A reminder message is shown at the beginning of the measurement (see Figure 1.106).

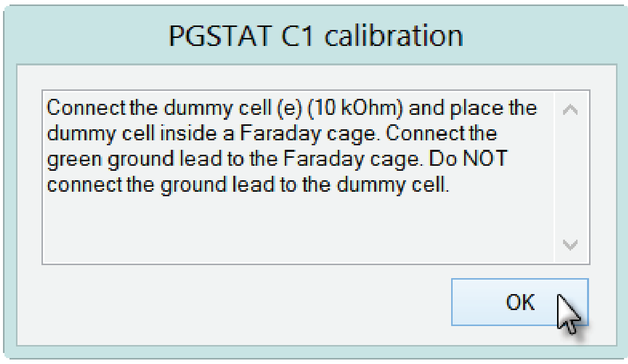


Figure 1.106 – A reminder message is shown at the beginning of the measurement

6. During the measurement, the measured data will be plotted as a Bode plot should be similar to the example shown in Figure 1.107.

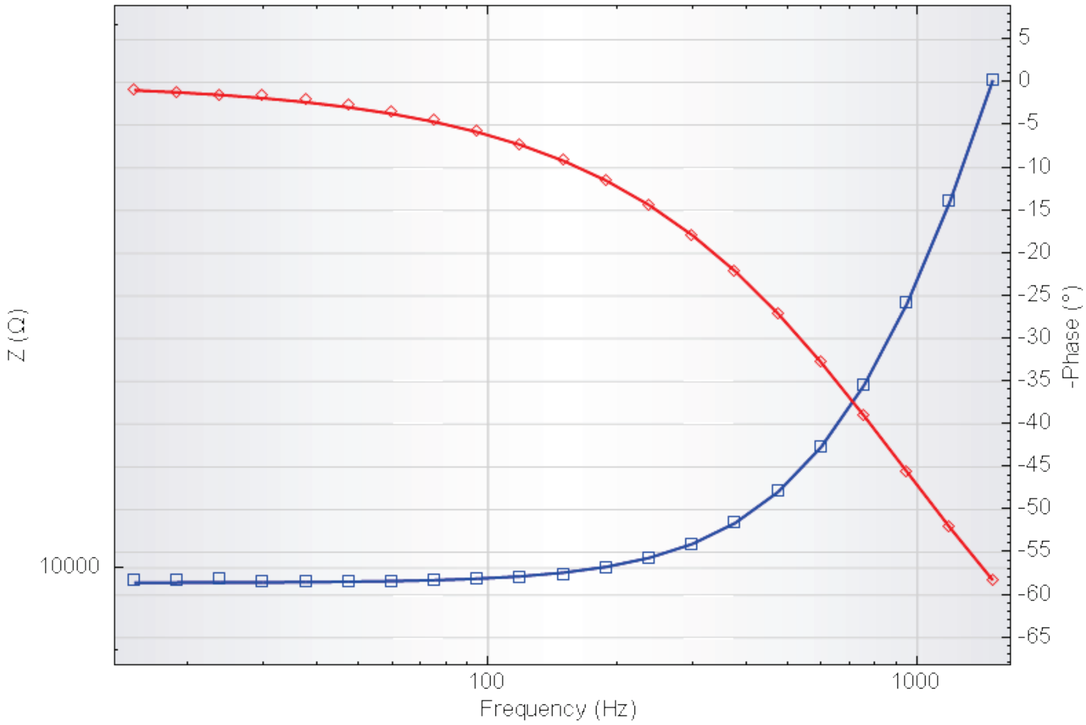


Figure 1.107 – Typical Bode plot obtained during the C1 calibration

7. The data is automatically fitted and the results of the fitting are reported in a Message box at the end of the measurement (see Figure 1.108).

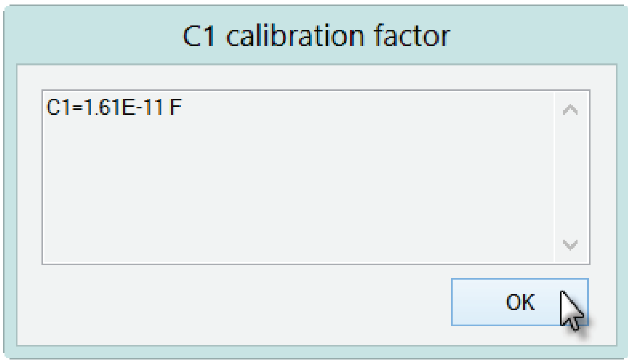


Figure 1.108 – The experimentally determined value of C1 is reported in a Message box at the end of the measurement

8. Open the Hardware setup of Nova (Tools – Hardware setup). Select the instrument type in the **Main Module** frame in the hardware setup window and adjust the value of C1 to the value reported in the Message box (see Figure 1.109).

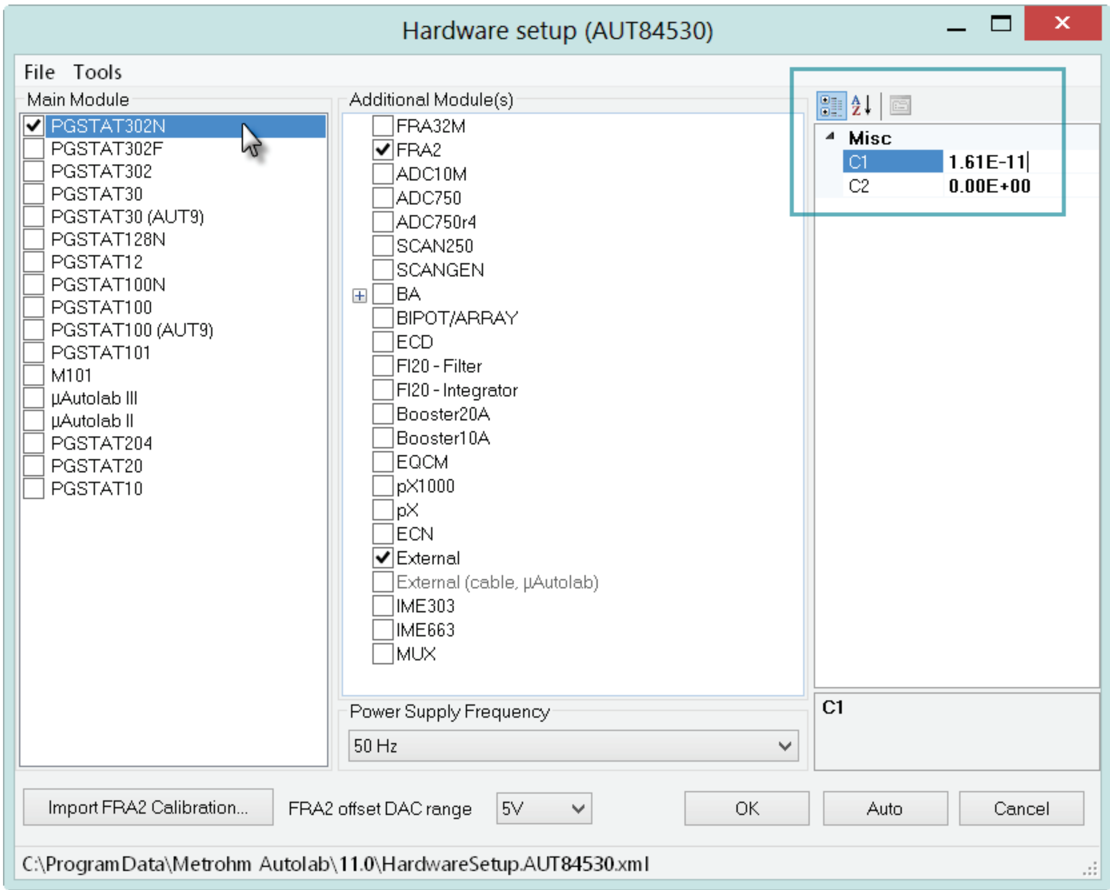


Figure 1.109 – Change the value of C1 to the value reported in the Message box

9. Click OK to save the changes and wait for the Autolab to be reinitialized using the updated Hardware setup.

1.6.18.2 – Determination of C2

Follow the steps described in this section to determine the value of the **C2** parameter.

1. Load the procedure *PGSTAT C2 calibration* from the *Module test* database.
2. Disconnect the Dummy cell and leave the leads open in the Faraday cage. CE and RE must be connected together as well as WE and S (as shown in Figure 1.110). Make sure RE/CE and WE/S are not connected together. Connect the ground lead from the PGSTAT to the Faraday cage.

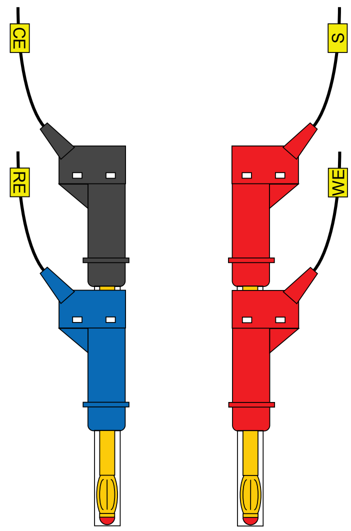


Figure 1.110 – Overview of the connection required for the determination of C2

3. Start the measurement and wait until it has been finished. Ignore the warning displayed during the procedure validation (see Figure 1.111).

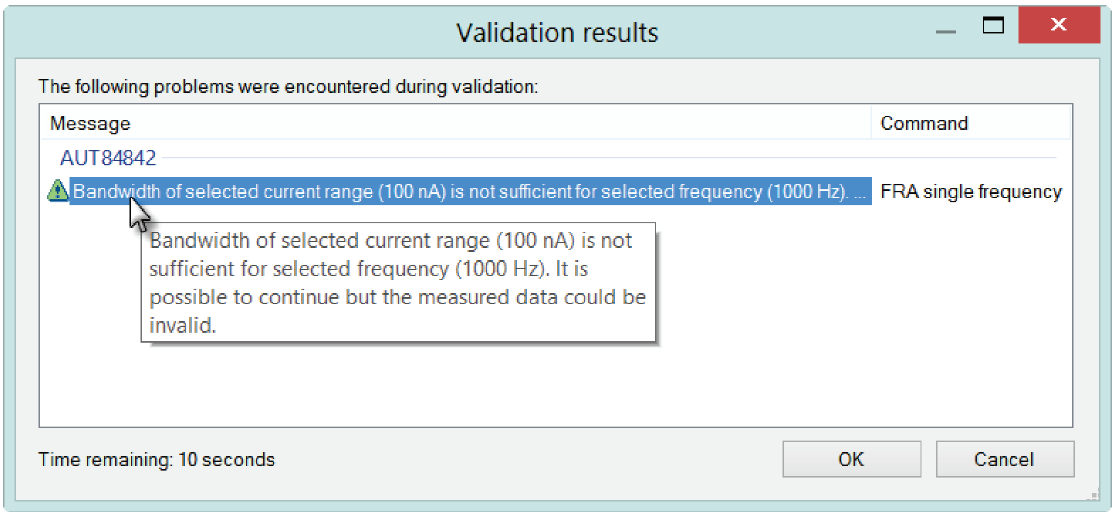


Figure 1.111 – Ignore the warning shown during the validation of the procedure

4. A reminder message is shown at the beginning of the measurement (see Figure 1.112).

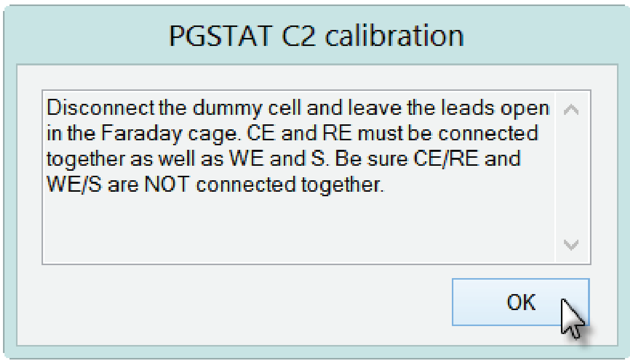


Figure 1.112 – A reminder message is shown at the beginning of the measurement

5. During the measurement, the measured data will be plotted as a Bode plot should be similar to the example shown in Figure 1.113.

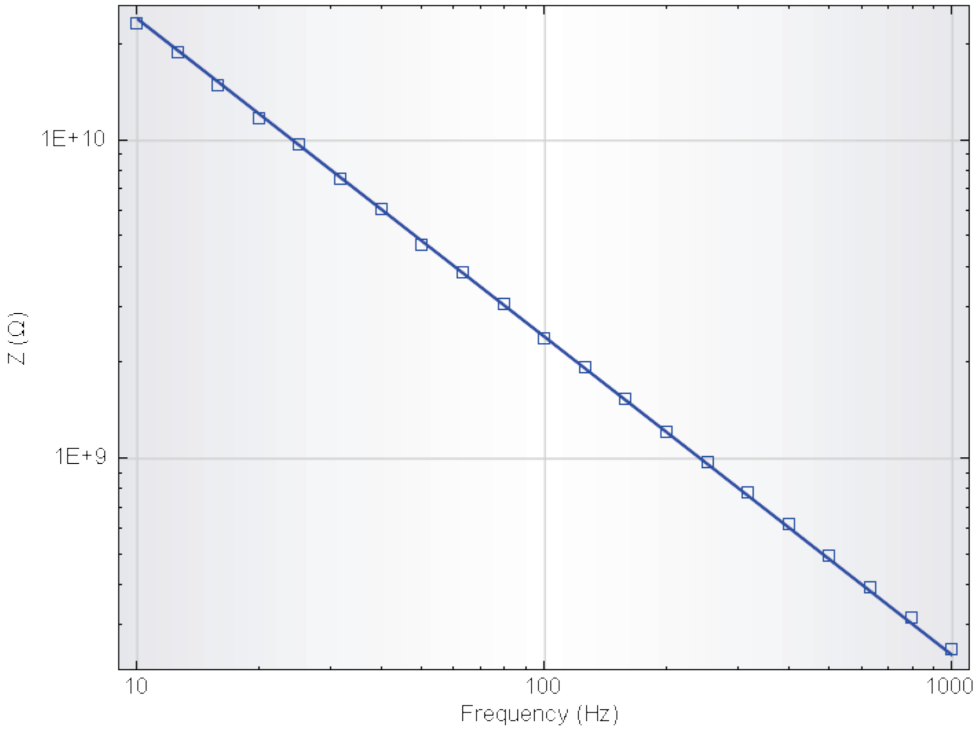


Figure 1.113 – Typical Bode plot obtained during the C2 calibration

6. The data is automatically fitted and the results of the fitting are reported in a Message box at the end of the measurement (see Figure 1.114).

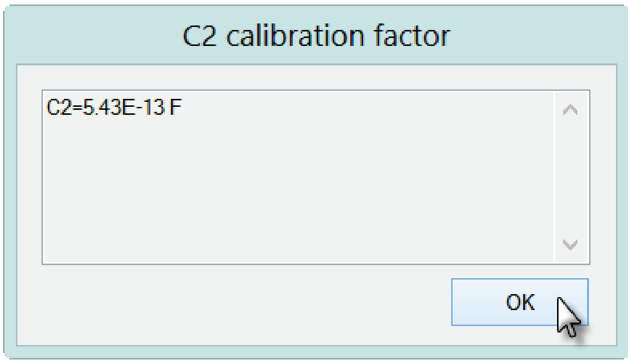


Figure 1.114 – The experimentally determined value of C2 is reported in a Message box at the end of the measurement

7. Open the Hardware setup of Nova (Tools – Hardware setup). Select the instrument type in the **Main Module** frame in the hardware setup window and adjust the value of C2 to the value reported in the Message box (see Figure 1.115).

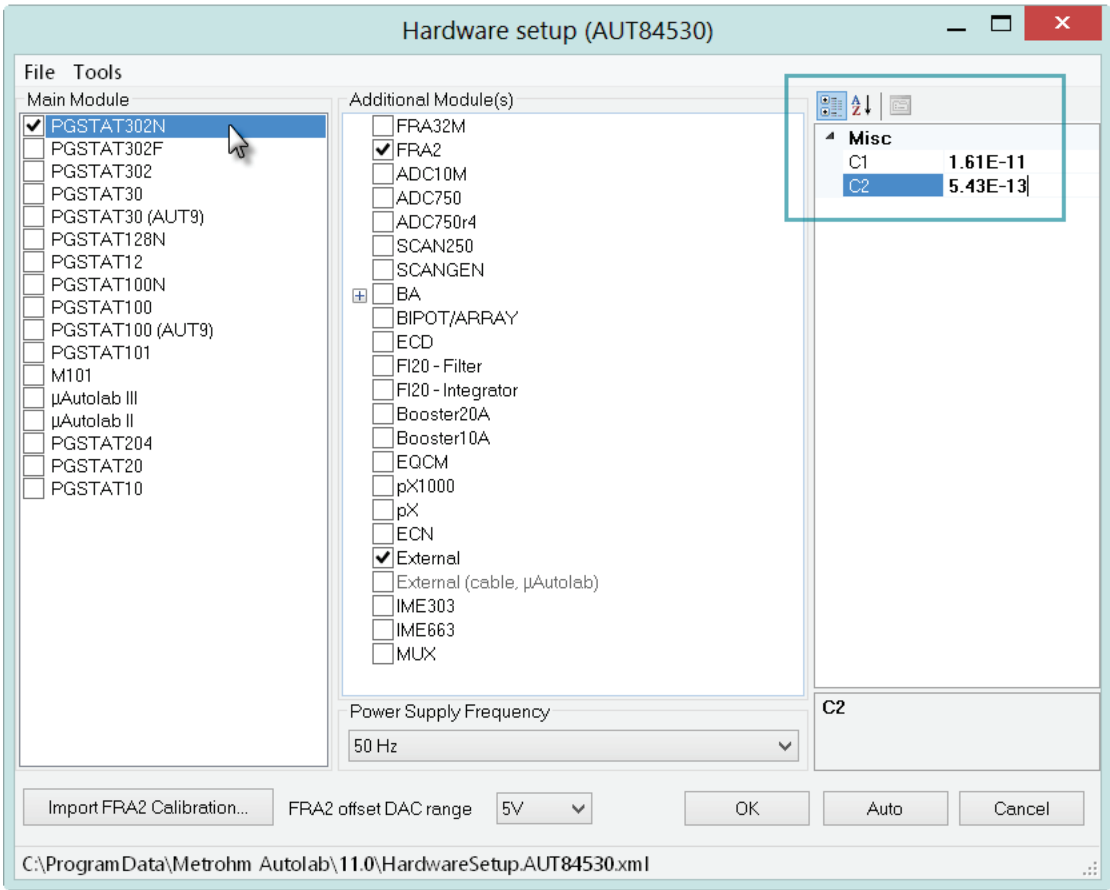


Figure 1.115 – Change the value of C2 to the value reported in the Message box

8. Click OK to save the changes and wait for the Autolab to be reinitialized using the updated Hardware setup.

Nova Getting started

The aim of the NOVA Getting started is to give new users a feel of the main features of the software as well as to introduce them to its mechanics. It is also intended to test the installation of the software. The example illustrated in this section will be presented without a specific clarification for each instruction or command. This chapter is meant to be used as a walkthrough for first time users. All of the aspects of the software are discussed in more detail in the User Manual. The document can be accessed in Nova by pressing the F1 key or through the Help menu.

2 – A typical Nova measurement

A typical Nova measurement starts with a procedure. This procedure must be selected, modified if necessary and executed. Nova will run through the instructions of the procedure and carry them out sequentially. While this happens, the collected data points will be displayed in real time. At the end of the measurement the data points will be available for further analysis.

Note

GPES users are used to start a measurement by selecting a pre-defined method from a list of available techniques. Nova is designed to perform complex measurements, seamlessly switching from one electrochemical method to another in a single procedure (see Chapter 2 of the User manual for further information). Therefore the electrochemical method selection becomes obsolete (ready to use template procedures are included, see Chapter 3 for more information).

For this quick start the Autolab is used in conjunction with the dummy cell.

2.1 – Starting up the software (installation required, see Chapter 1)

Nova can be started by double clicking the Nova shortcut on the computer desktop. If an Autolab is already connected to the computer through the USB connection and turned on, the software will automatically identify the instrument and upload the required control software.

If no instrument is connected after starting Nova, connecting the Autolab to the computer using the USB and turning it on will trigger the initialization process automatically (see Chapter 1 for further details on the USB communication with the instrument).

By default, Nova will start in the **Setup view**. The Setup view is one of the four views the user can select while operating Nova. The other three are the Measurement view (used to display the data in real time during a measurement),

the Analysis view (used to perform data analysis) and the Multi Autolab view (used to control the Multi Autolab or more than one instrument at the same time).

The Setup view contains several areas (also called frames). Figure 2.1 shows an overview of the Setup view.

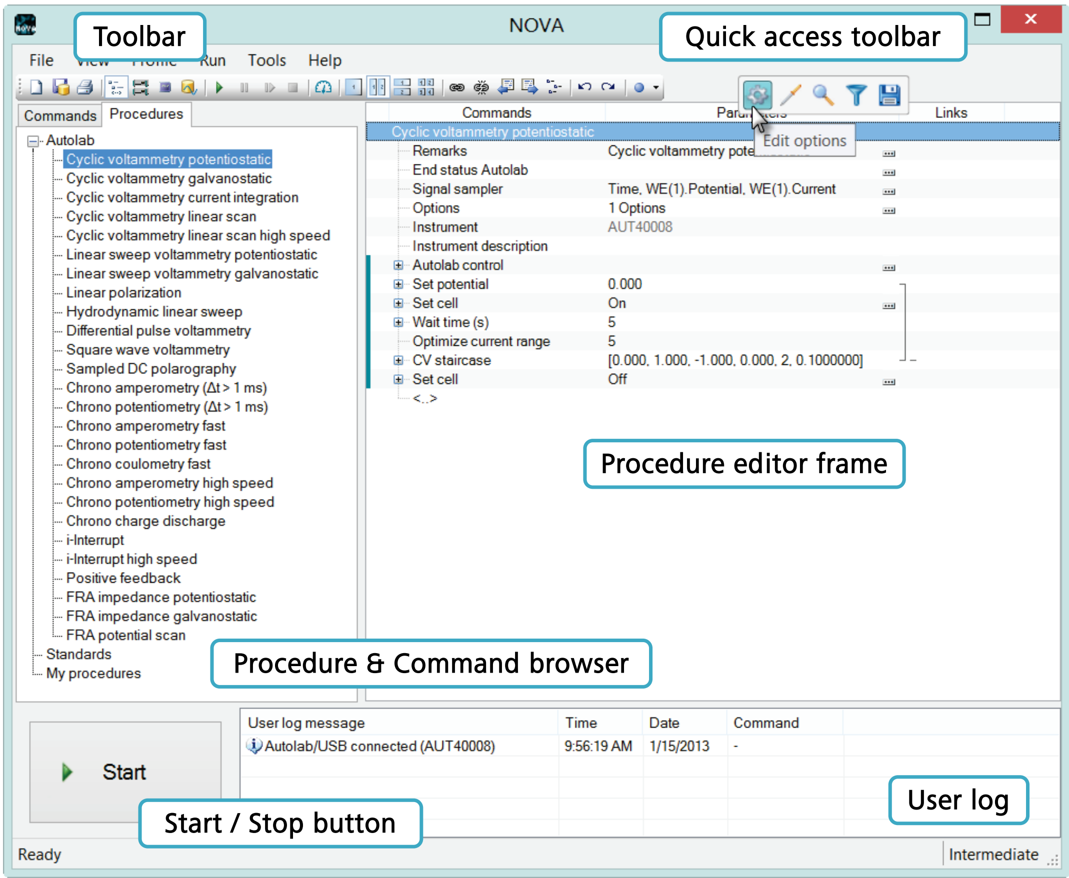


Figure 2.1 – Overview of the Setup view of Nova

More information regarding the Setup view of Nova can be found in Chapter 2 of the User Manual.

The procedure browser frame displays a number of available procedures, in the Autolab group. Figure 2.2 shows a more detailed view of the Setup view.

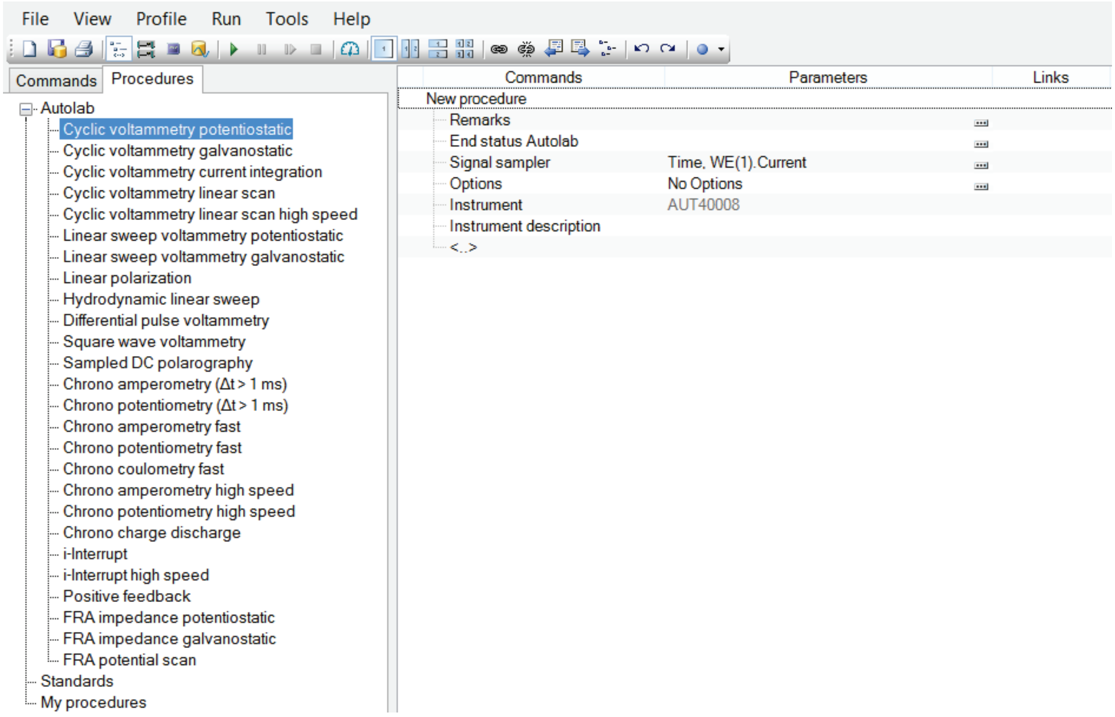


Figure 2.2 – Details of the Setup view

The procedures visible in the Autolab group in the browser are standard factory procedures. These procedures are always visible and cannot be changed or removed.

Note

The actual number of procedures listed in the Autolab group of procedures in the Setup view depends on the active profile. By default, the Cyclic voltammetry potentiostatic procedure should be visible in this group, unless it has been hidden on purpose. Please refer to the User manual for more information on the use of profiles in Nova.

2.2 – Running cyclic voltammetry on the dummy cell

The purpose of this quick start is to perform staircase cyclic voltammetry on the Autolab dummy cell. In the example discussed below, the dummy cell (c) is used. The cell cables should therefore be connected to the dummy cell as displayed in Figure 2.3.

Note

The PGSTAT101 is not equipped with the Autolab dummy cell. An optional external dummy cell can be obtained (Contact your Autolab distributor for more information). For the PGSTAT101, use the procedure TestCV PGSTAT101 available in the Module test database (Refer to the Module test with Nova document, available from the Help – Tutorials menu).

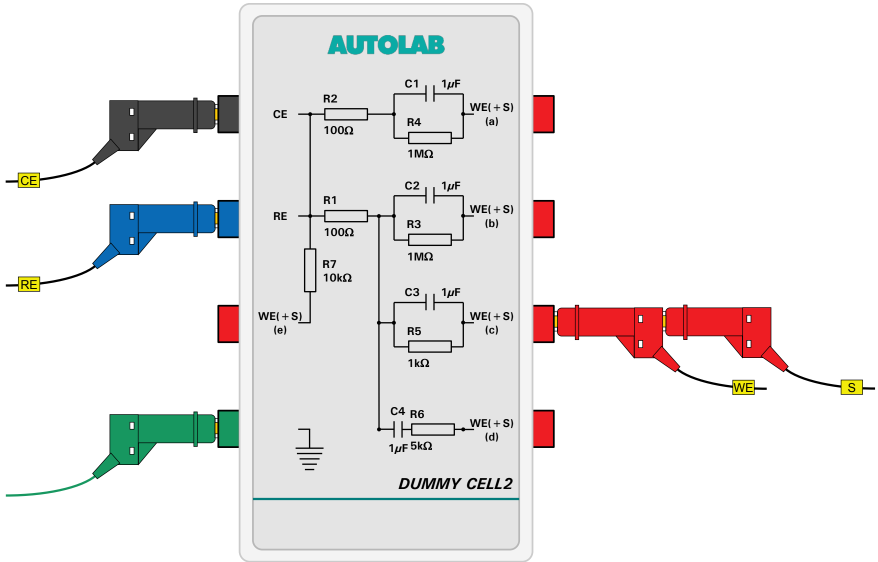


Figure 2.3 – Dummy cell connections

2.2.1 – Setting up the experiment

To perform a cyclic voltammetry experiment, the default Cyclic voltammetry potentiostatic procedure must be loaded into the Procedure editor. Right clicking the Cyclic voltammetry potentiostatic procedure in the browser brings up a context menu, displaying the *Open for editing* option (see Figure 2.4).

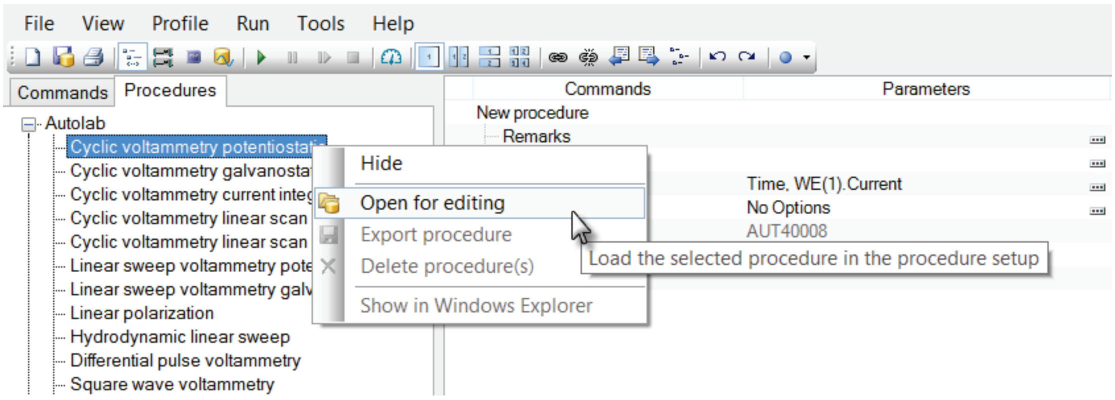


Figure 2.4 – Loading a procedure in the Editor frame (part 1)

Clicking this instruction will load the procedure in the editor frame. The name of procedure will change from New procedure to Cyclic voltammetry potentiostatic. A series of **commands** will be displayed under the Cyclic voltammetry potentiostatic procedure in the editor frame (see Figure 2.5).

These commands form the procedure and will be executed sequentially when the procedure is started.

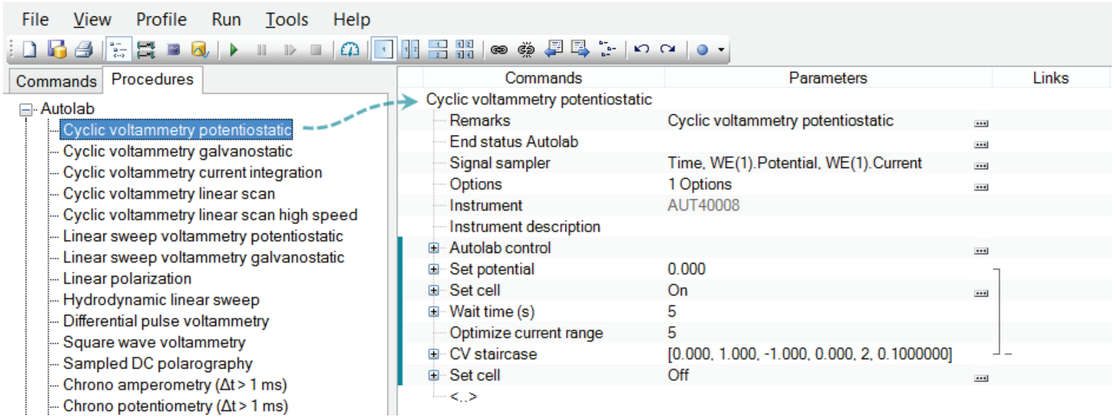


Figure 2.5 – Loading a procedure in the Editor frame (part 2)

Once the procedure is loaded in the procedure editor frame, it can be executed. This procedure will perform a single potential scan, between -1 V and 1 V on the dummy cell, starting at a potential of 0 V, with a scan rate of 100 mV/s.

Note

In Nova, a procedure is defined as the combination of a **signal sampler** and a series of **commands**. The signal sampler defines which signals (current, potential, time, pH, …) will be sampled during the measurement and the commands define how these signals will be sampled.

When the procedure is loaded in the procedure editor frame it can be modified and started. It is convenient to name each experiment in a unique way, for bookkeeping purposes. To change the name of the cyclic voltammetry potentiostatic procedure to a custom name, click the cyclic voltammetry potentiostatic name in the procedure editor and change it to *Quick start Cyclic voltammetry* (see Figure 2.6).

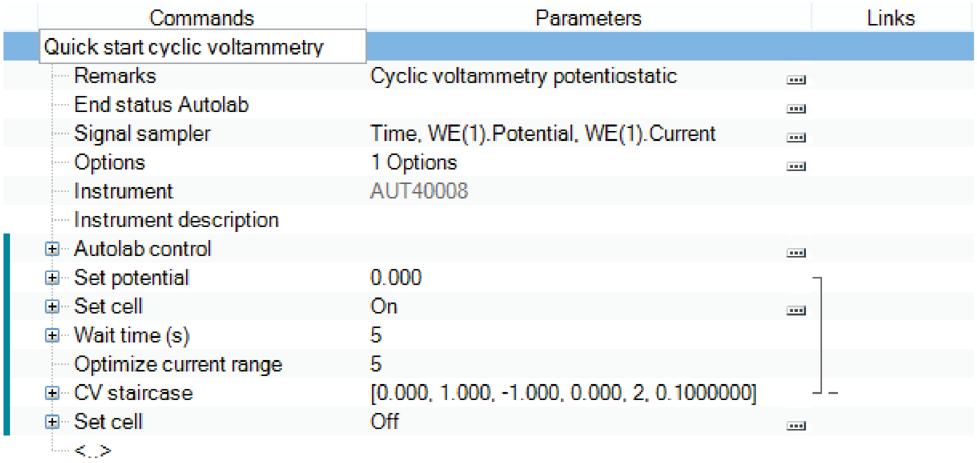


Figure 2.6 – Editing the procedure name

After the title has been edited, validate with the Enter key and save the procedure, using the File menu – Save procedure as New (see Figure 2.7).

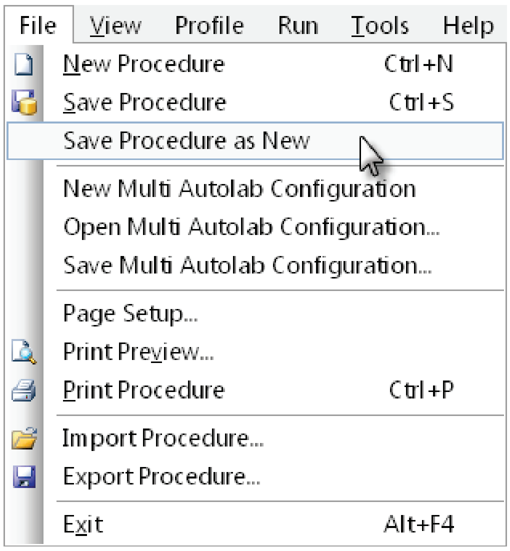


Figure 2.7 – Save the procedure

The procedure will be added to the My procedures database (see Figure 2.8).

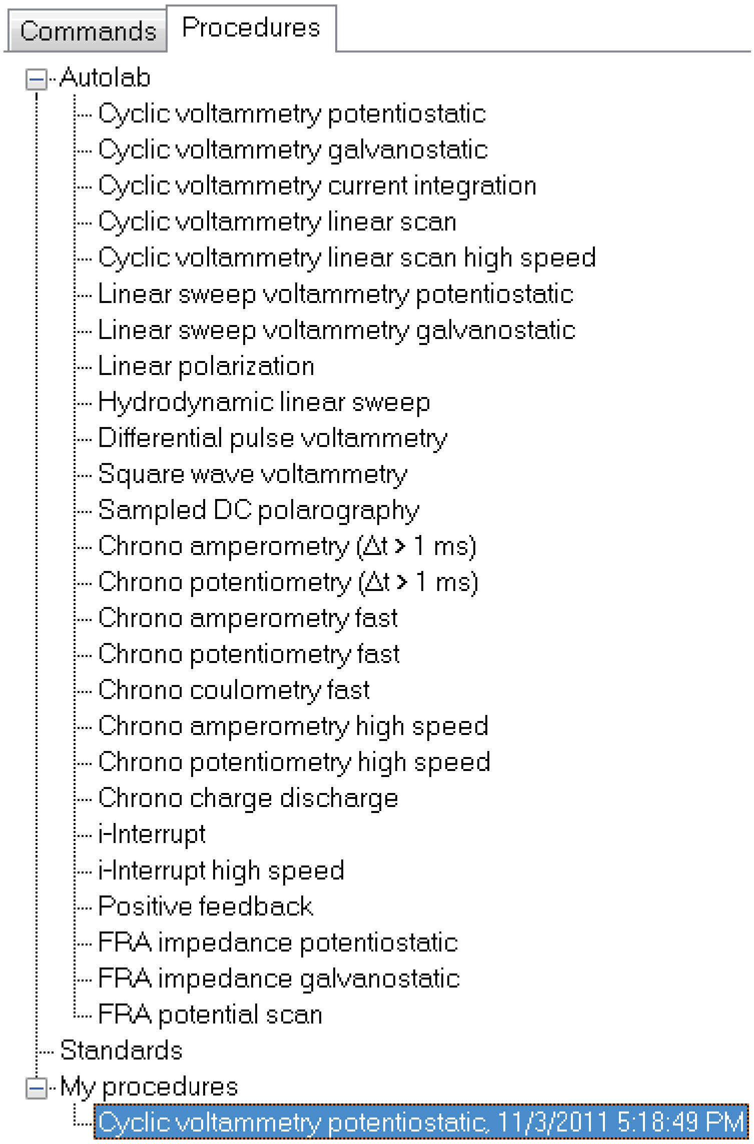


Figure 2.8 – Adding the quick start cyclic voltammetry procedure

The procedure can now be started. Click the Start button located at the bottom of the screen to begin the experiment. The procedure is first validated, which can take a few seconds depending on the amount of commands in the procedure. If no errors are detected, the measurement starts. The software will automatically switch to the Measurement view where the measured data points are displayed in real time.

You can also switch to the Measurement view at any time by clicking the measurement view button in the toolbar (see Figure 2.9).

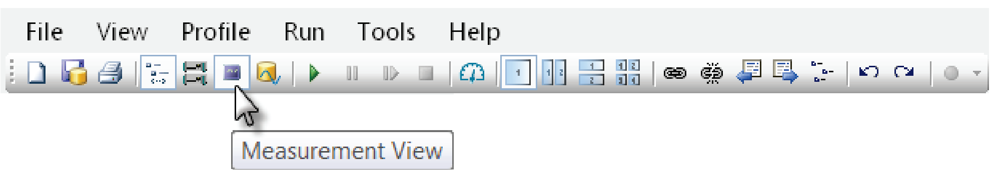


Figure 2.9 – Switching from the setup view to the measurement view

2.2.2 – Viewing the measured data

The **measurement view** displays the measured data in real time. The default display settings for a cyclic voltammetry experiment are the potential (Potential applied) on the X-axis and the measured current on the Y-axis (WE(1).Current). The scale of the plot is automatically adjusted during the measurement.

When the measurement is running, the **start button** is replaced by a **stop button** that can be pressed to abort the experiment. Figure 2.10 shows the measurement view during the Quick start experiment.

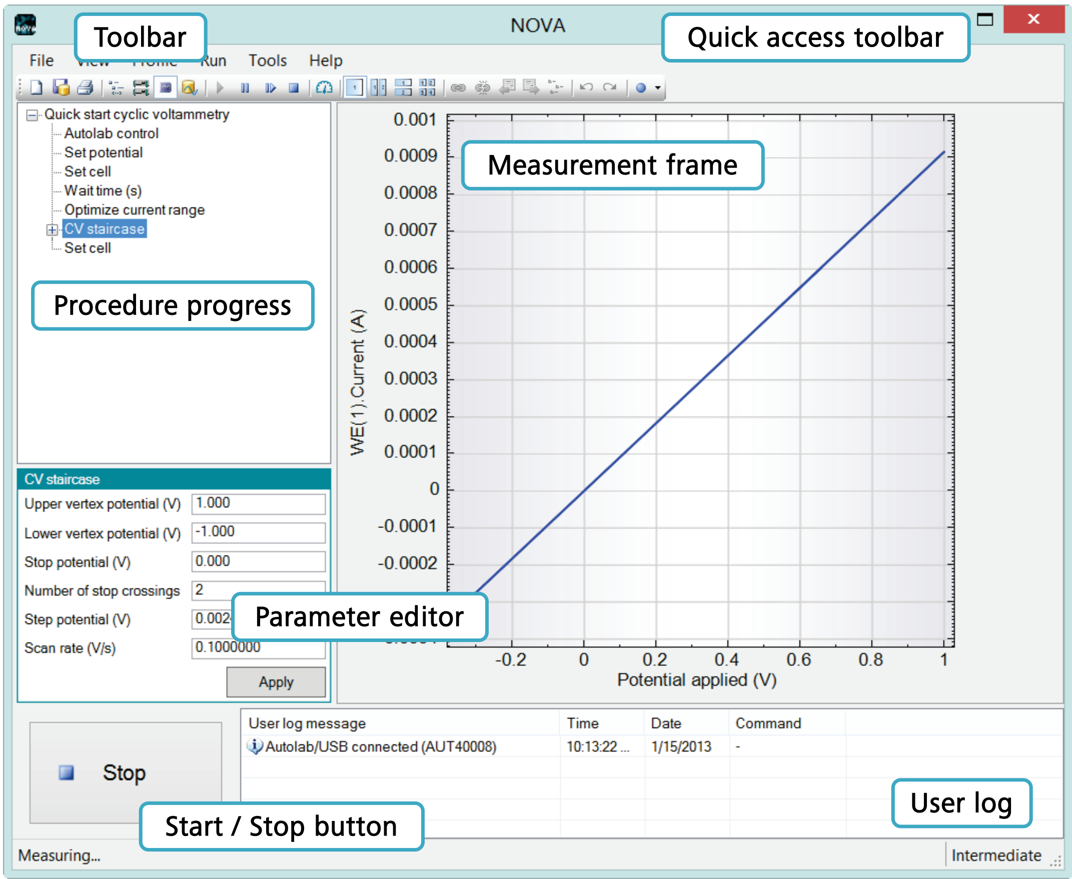
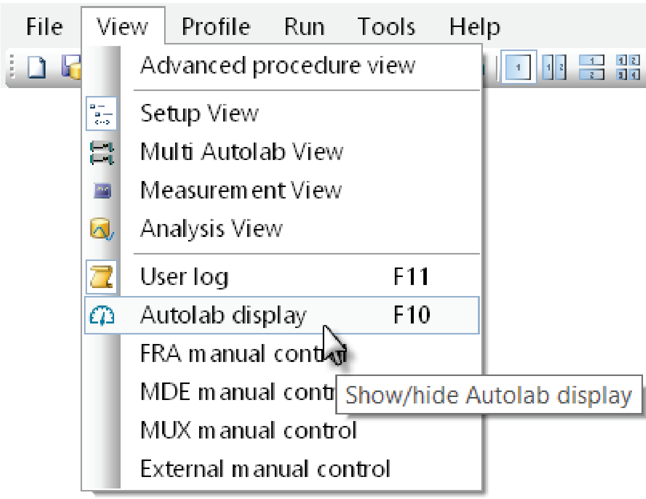


Figure 2.10 – The Measurement view

It is possible, at any time, to pick the Autolab display option from the View menu (or to press the F10 shortcut key), as shown in Figure 2.11.



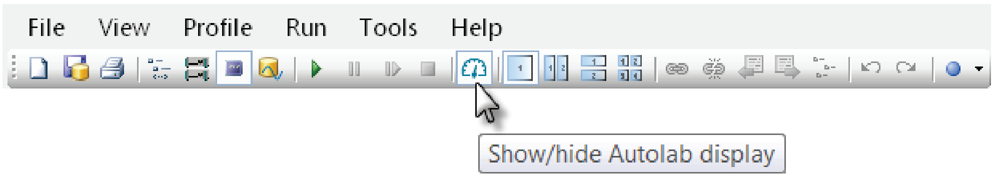


Figure 2.11 – Select the Autolab display option in the View menu or use the dedicated button in the toolbar to show or hide the Autolab display window

Figure 2.12 shows the Autolab display during the measurement.

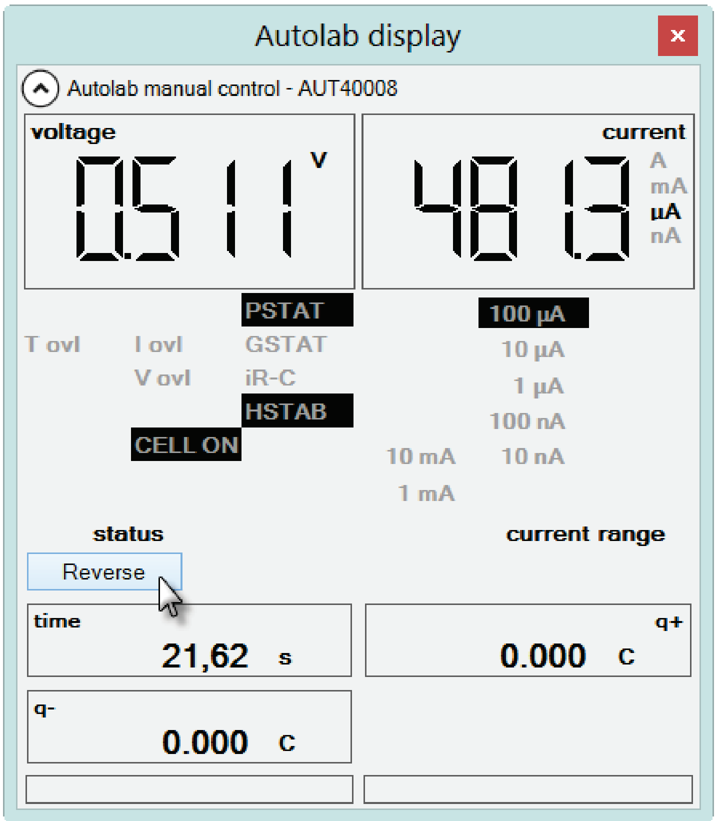


Figure 2.12 – The Autolab display

The Autolab display provides real-time information about the sampled signals and the hardware settings and provides additional controls, like the Reverse button, which can be used to reverse the scan direction[21].

The procedure used in this quick start guide performs a single scan on the dummy cell. When the scan is finished, the stop button becomes a start button again, indicating that Nova is ready to perform a new measurement.

At the end of the measurement, the User log can be updated, depending on the events that occurred during the measurement. For example, if a current overload occurred during the experiment, a message will be shown in the log (see Figure 2.13).

User log message	Time	Date	Command
Autolab/USB connected (AUT40008)	10:20:35 AM	1/15/2013	-
Overload occurred in 1 µA current range, use a higher current range.	10:23:18 AM	1/15/2013	CV staircase

Figure 2.13 – The User log is automatically updated at the end of the experiment

Although the measurement view displays the measured data during and after the experiment, it is not meant for **data analysis**. Data analysis is performed in the dedicated **analysis view**. Switching to the analysis view can be done by clicking the corresponding button on the toolbar (see Figure 2.14).

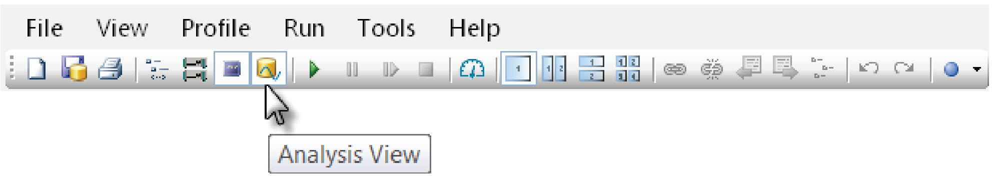


Figure 2.14 – Switching from the measurement view to the analysis view

2.2.3 – Analyzing the measured data

The analysis view is used to manage experimental data and perform data analysis. Figure 2.15 shows the default layout.

[21] Please refer to the Cyclic voltammetry tutorial, available from the Help – Tutorials menu, for more information.

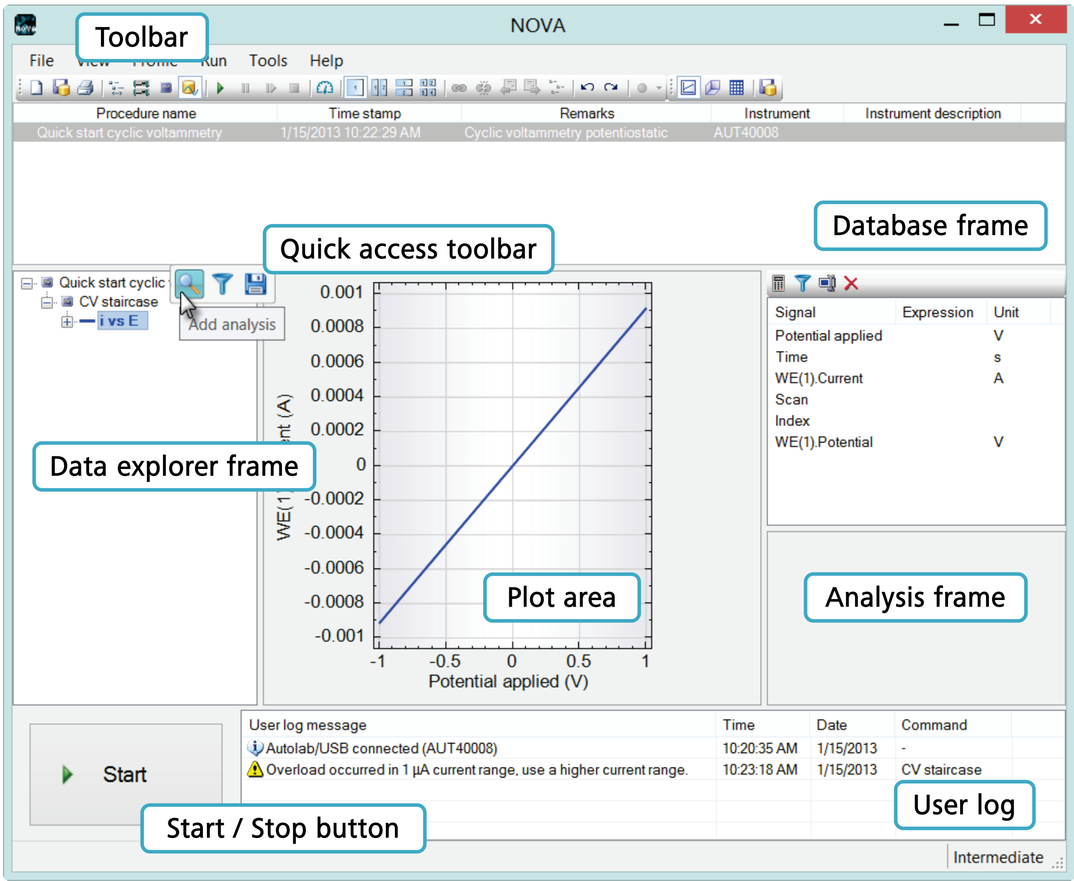


Figure 2.15 – The analysis view

The analysis view has several noteworthy features, the most important of which is the **database.** Every measurement is stored in the database automatically. Each entry of the database corresponds to a measurement and is logged together with the time and date, as well as a Remarks field and the serial number of the instrument used in the experiment. An additional field, Instrument description can be used to provide a description of the instrument (see Figure 2.16).

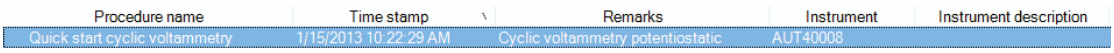


Figure 2.16 – Database entries are logged by Procedure name, Time stamp, Remarks, Instrument and Instrument description

The database consists of one single folder. However, if required, a specific entry of the database can be exported as a single file[22].

[22] Please refer to the User Manual for more information.

The analysis view features a dedicated toolbar (see Figure 2.17).

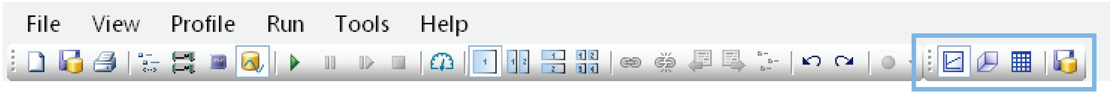


Figure 2.17 – The analysis view toolbar (highlighted)

To view and analyze the data from a measurement, the corresponding entry of the database has to be loaded in the Data explorer frame.

Double click the *Quick start Cyclic voltammetry* entry of your database to load it in the data explorer frame. The database entry will appear in this frame as shown in Figure 2.18.

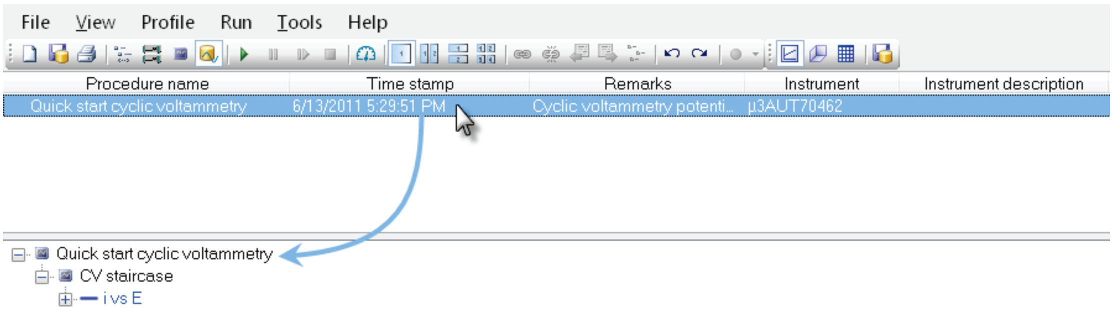


Figure 2.18 – Loading the measured data in the data explorer

Once the data from the database has been loaded into the data explorer frame, it is available for data analysis. To view the data, click the blue **i vs E** item in the data explorer. The measured data will be displayed using the default settings, i.e., plotting the Potential applied on the X-axis and the measured current, WE(1).Current on the Y-axis. The measured data should be displayed in the data analysis frame like in Figure 2.19.

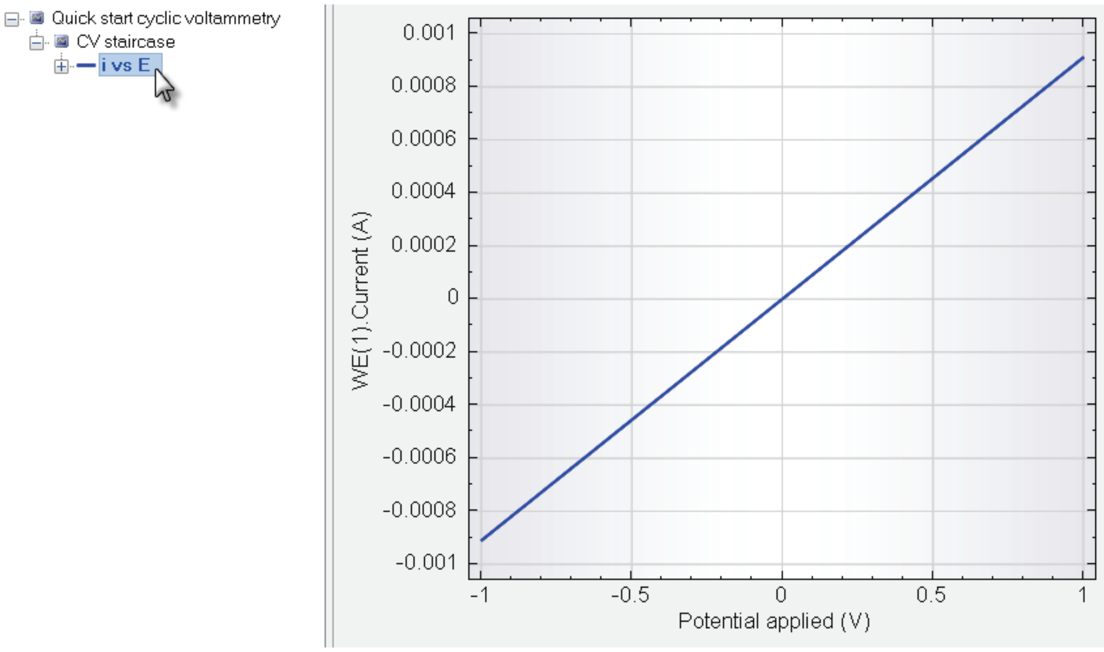


Figure 2.19 – Displaying the measured data in the analysis view

The final part of this quick start guide will illustrate some of the features of the analysis view. More information can be found in Chapter 4 of the User Manual.

During this experiment, the Autolab instrument recorded values for time, current and potential. These experimental values are known in Nova as **Signals**. These signals can be used in any combination to control the way the data is plotted.

Click the ⊞ symbol next to the blue i vs E line in the data explorer frame to reveal the signals currently used for this plot (see Figure 2.20).

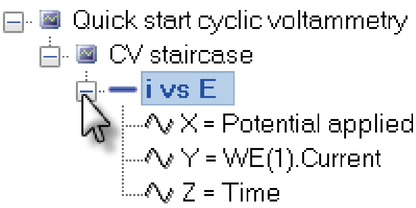


Figure 2.20 – Expanding the Signal set line in the data explorer frame

Figure 2.20 shows that for the current plot, the Potential applied signal is used for the X-axis and the WE(1).Current signal is used for the Y-axis. The WE(1).Current signal used for the Z-axis is not relevant for a 2D plot.

It might be useful to show the applied potential (on the Y-axis) as a function of time (on the X-axis). This can be easily done within the analysis view by right clicking the active setting for the X-axis (the WE(1).Potential applied) in the data explorer frame and replacing it by the time. The same can be done to change the signal plotted on the Y-axis from the measured WE(1).Current to the WE(1).Potential (see Figure 2.21).

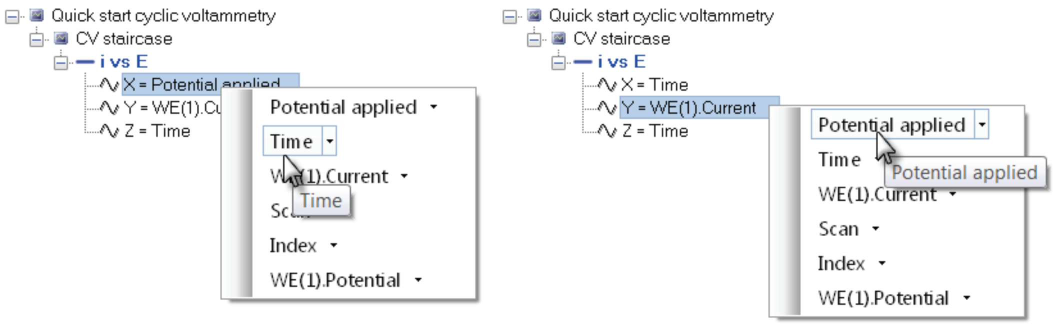


Figure 2.21 – Changing the plot settings

After changing these settings, the plot should be similar to Figure 2.22.

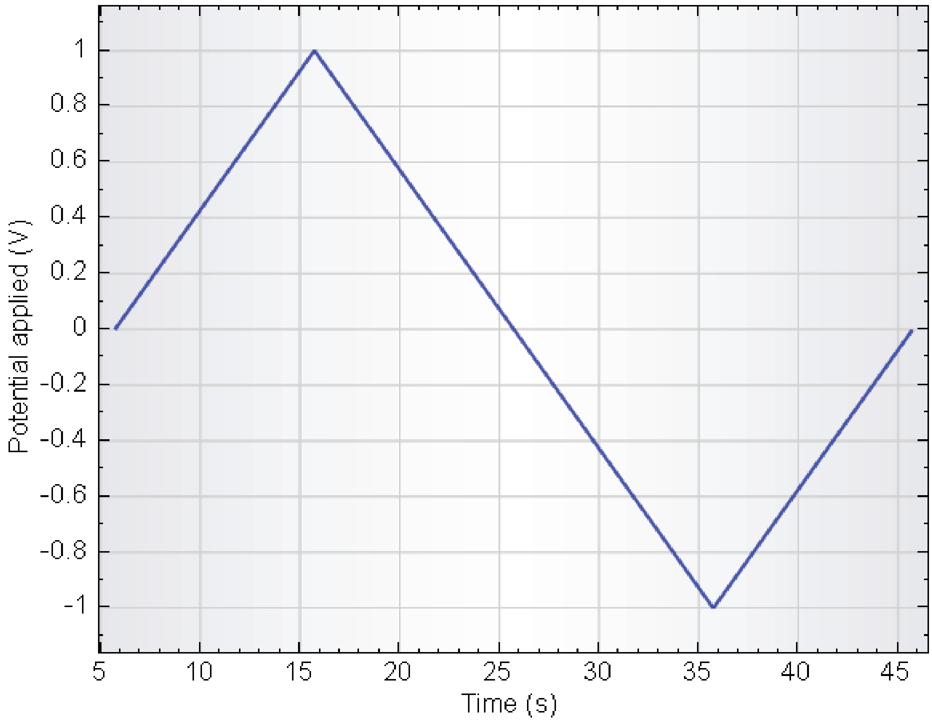


Figure 2.22 – Plotting the applied potential as a function of time

The familiar saw-tooth profile of a cyclic voltammogram can be easily recognized.

Nova also provides a 3D plot engine. To switch to a 3D plot, click the corresponding button  of the data analysis view toolbar (see Figure 2.23).

Figure 2.23 – Showing the data in 3D

The 3D plot displays time, current, and potential on the same plot (use the WE(1).Current as the Signal for the Z-axis). This plot can be turned and rotated by

clicking the graph and moving the mouse around while holding the left button (see Figure 2.24).

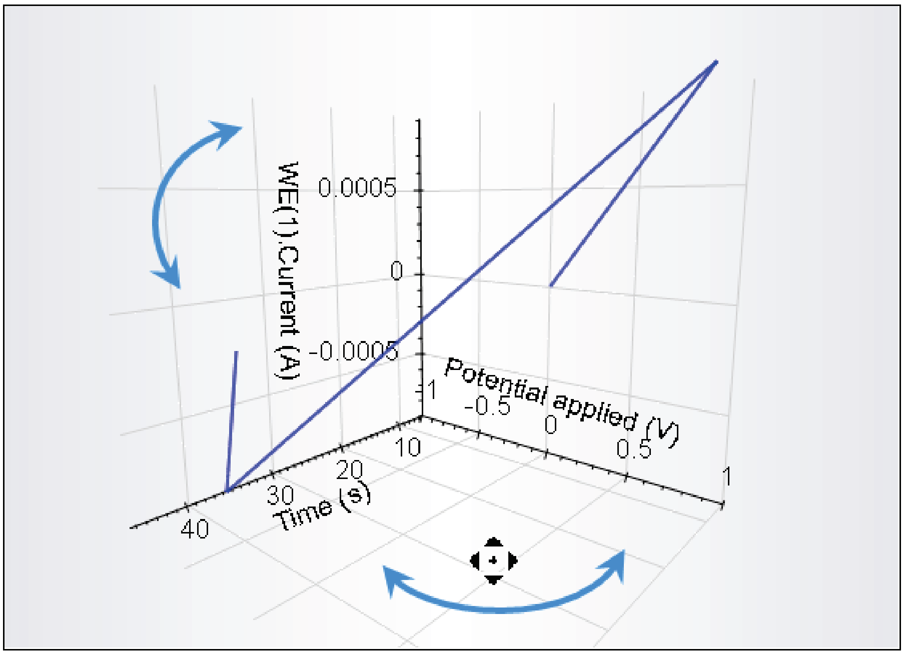


Figure 2.24 – Spinning the 3D graph around

Note

While holding the left mouse button, the mouse pointer changes to the pointer highlighted in Figure 2.24.

Feel free to try to change the plot, either in the 2D or the 3D view. We recommend that you take the time to get familiar with the Nova basics before exploring the rest of the manual for more information.

2.2.4 – Using the data grid

A very important feature of Nova is the **Data grid** and its functionality. During a measurement, several signals are sampled and are stored in the database when the measurement is completed. These signals are then available in the analysis view for plotting purposes, as shown in the previous section.

For the standard Cyclic voltammetry potentiostatic measurement, these signals are:

- Potential applied
- WE(1).Current
- WE(1).Potential
- Scan
- Time
- Index

The data grid provides an overview of all the signals. To access the data grid, click the corresponding button, , in the toolbar (see Figure 2.25).

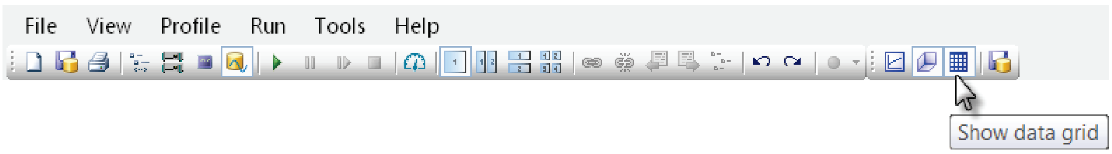


Figure 2.25 – Selecting the data grid

The data grid displays all the values of each signal that was recorded during the measurement. Scrolling down the list allows you to inspect all the data points (see Figure 2.26).

Potential applied (V)	WE(1).Potential (V)	WE(1).Current (A)	Time (s)	Scan	Index
0	0.000378418	5.09247E-7	5.5693	1	1
0.00244141	0.00282898	1E-6	5.59372	1	2
0.00488281	0.00525818	4.96094E-6	5.61813	1	3
0.00732422	0.00771179	7.18597E-6	5.64255	1	4
0.00976563	0.0101563	9.41772E-6	5.66696	1	5
0.012207	0.0125549	1.16486E-5	5.69137	1	6
0.0146484	0.0150482	1.38672E-5	5.71579	1	7
0.0170898	0.0174652	1.6098E-5	5.7402	1	8
0.0195313	0.0198853	1.83289E-5	5.76462	1	9
0.0219727	0.0223602	2.05414E-5	5.78903	1	10

Figure 2.26 – The data grid displays the values of the signals

Using the data grid, it is possible to export the measured data points to other software's for data analysis (Excel, Origin, SigmaPlot, …). This can be done by right-clicking the data grid and by choosing the Export ASCII data option from the context menu (see Figure 2.27).

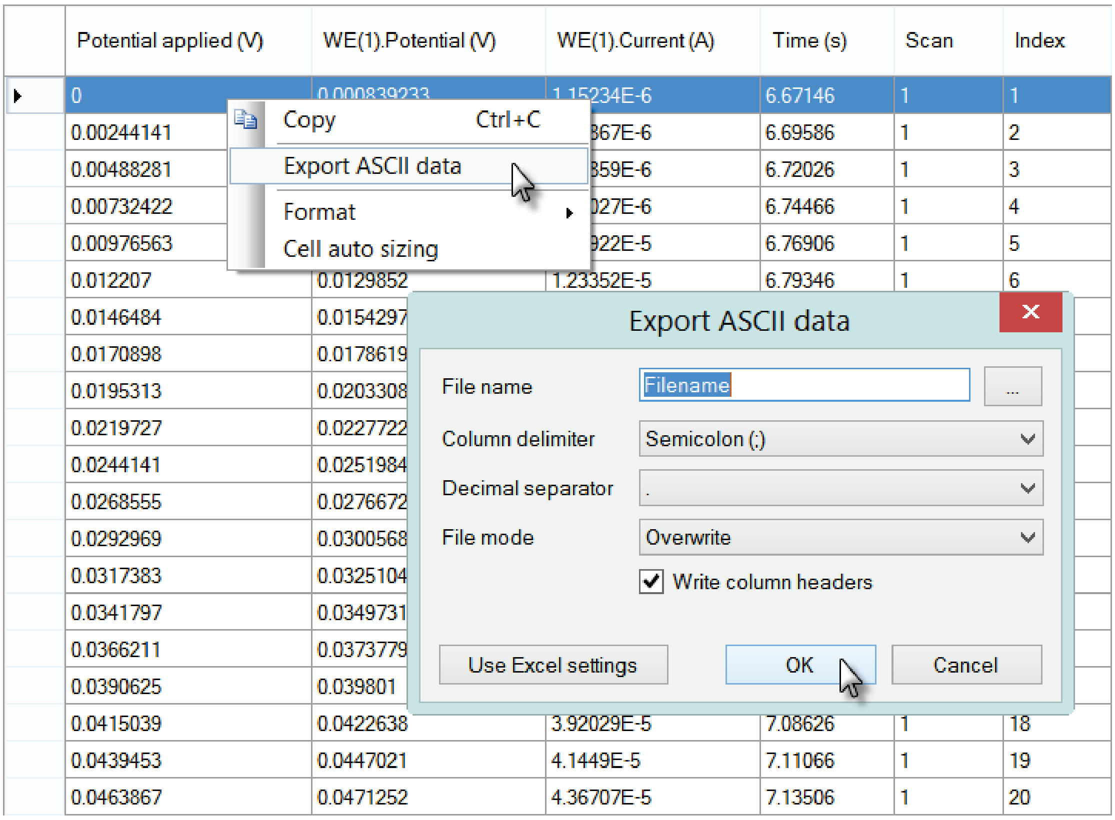


Figure 2.27 – Exporting the data to ASCII or Excel

It is also possible to create new signals based on calculations performed on the existing signals. For example, it can be useful to calculate the **logarithm** of the measured current.

The data grid can be used like a spreadsheet. It comes with a signal calculator which can be used to create a new signal based on an existing signal and a mathematical operation.

To create a new signal, click the CV staircase item in the data explorer frame and select the Calculate signal tool from the quick access toolbar by clicking the button (see Figure 2.28).

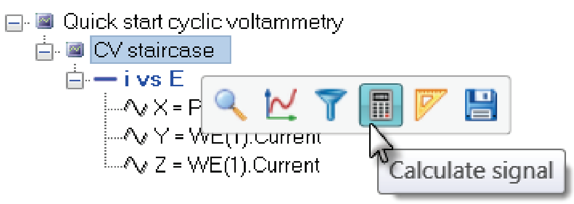
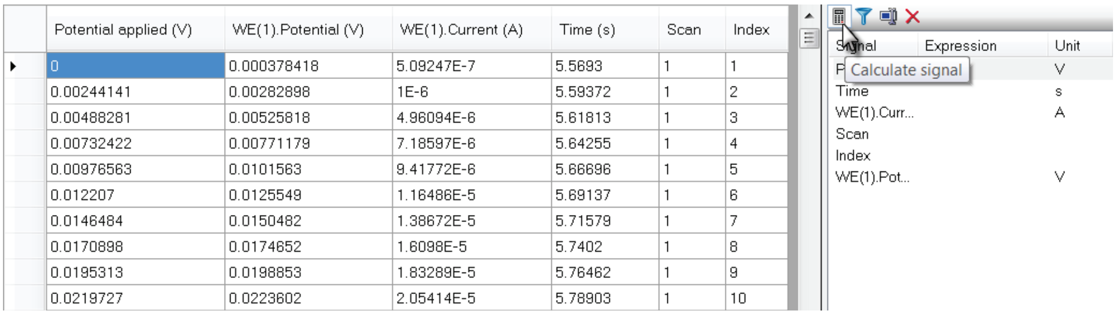


Figure 2.28 – Adding a calculate signal to the CV staircase

Alternatively, it is also possible to click the button located in the toolbar in the frame on the right-hand side of the data grid (see Figure 2.29).



	Potential applied (V)	WE(1).Potential (V)	WE(1).Current (A)	Time (s)	Scan	Index
▸	0	0.000378418	5.09247E-7	5.5693	1	1
	0.00244141	0.00282898	1E-6	5.59372	1	2
	0.00488281	0.00525818	4.96094E-6	5.61813	1	3
	0.00732422	0.00771179	7.18597E-6	5.64255	1	4
	0.00976563	0.0101563	9.41772E-6	5.66696	1	5
	0.012207	0.0125549	1.16486E-5	5.69137	1	6
	0.0146484	0.0150482	1.38672E-5	5.71579	1	7
	0.0170898	0.0174652	1.6098E-5	5.7402	1	8
	0.0195313	0.0198853	1.83289E-5	5.76462	1	9
	0.0219727	0.0223602	2.05414E-5	5.78903	1	10

Figure 2.29 – Opening the Calculate signal tool

The Calculate signal window will be displayed (see Figure 2.30).

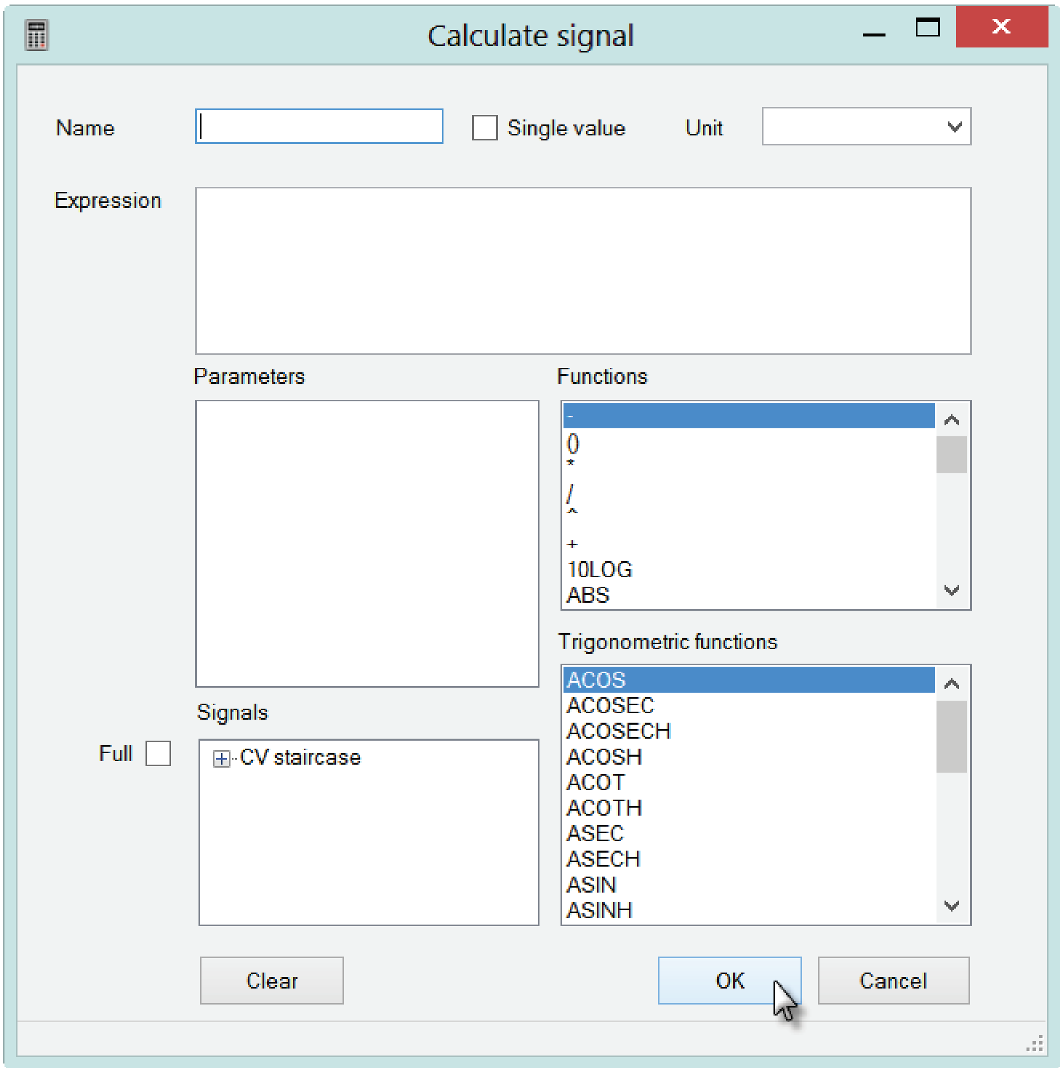


Figure 2.30 – The Calculate signal window

The calculate signal window works as an electrochemical calculator. It has several fields which are used to create a new signal:

- **Name:** this is the name of the new signal (a name is mandatory)
- **Single value:** this checkbox can be used to force the calculate signal to return a single value
- **Unit:** the unit of the new signal
- **Expression:** this is the mathematical expression used to calculate the new signal
- **Parameters:** a list of identified parameters used in the expression
- **Functions:** a list of common mathematical functions that can be used to calculate the new signal
- **Trigonometric functions:** a list of common trigonometric functions
- **Signals:** this is a list of the available signals in the data set

As an example, we are going to calculate the logarithm of the current in order to create a Tafel plot.

In the calculate signal window, type log(i) as a name to identify the new signal. Then, scroll down the list of functions to locate the **10LOG** function and double click it to add it to the expression builder (see Figure 2.31).

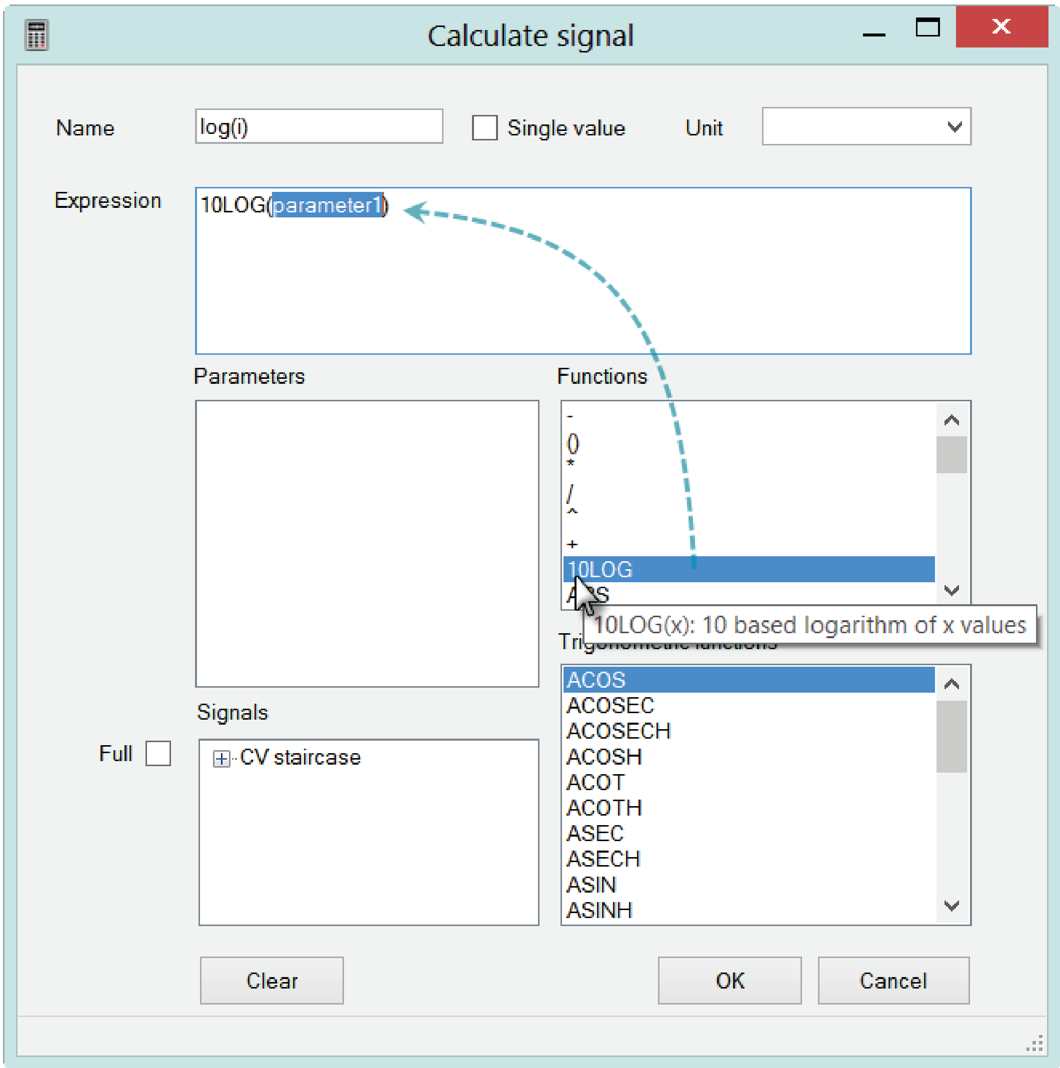


Figure 2.31 – Creating the log(i) signal – part 1: Defining the expression

Next, double click the **ABS** function, located under the **10LOG** function, in order to add it to the expression. Finally, in the expression, change the **[parameter1]** text to **i** and click the parameters frame in the expression builder. The expression builder will identify the parameter, **i**, as the only parameter of the expression. This parameter will be displayed in the Parameters frame (see Figure 2.32).

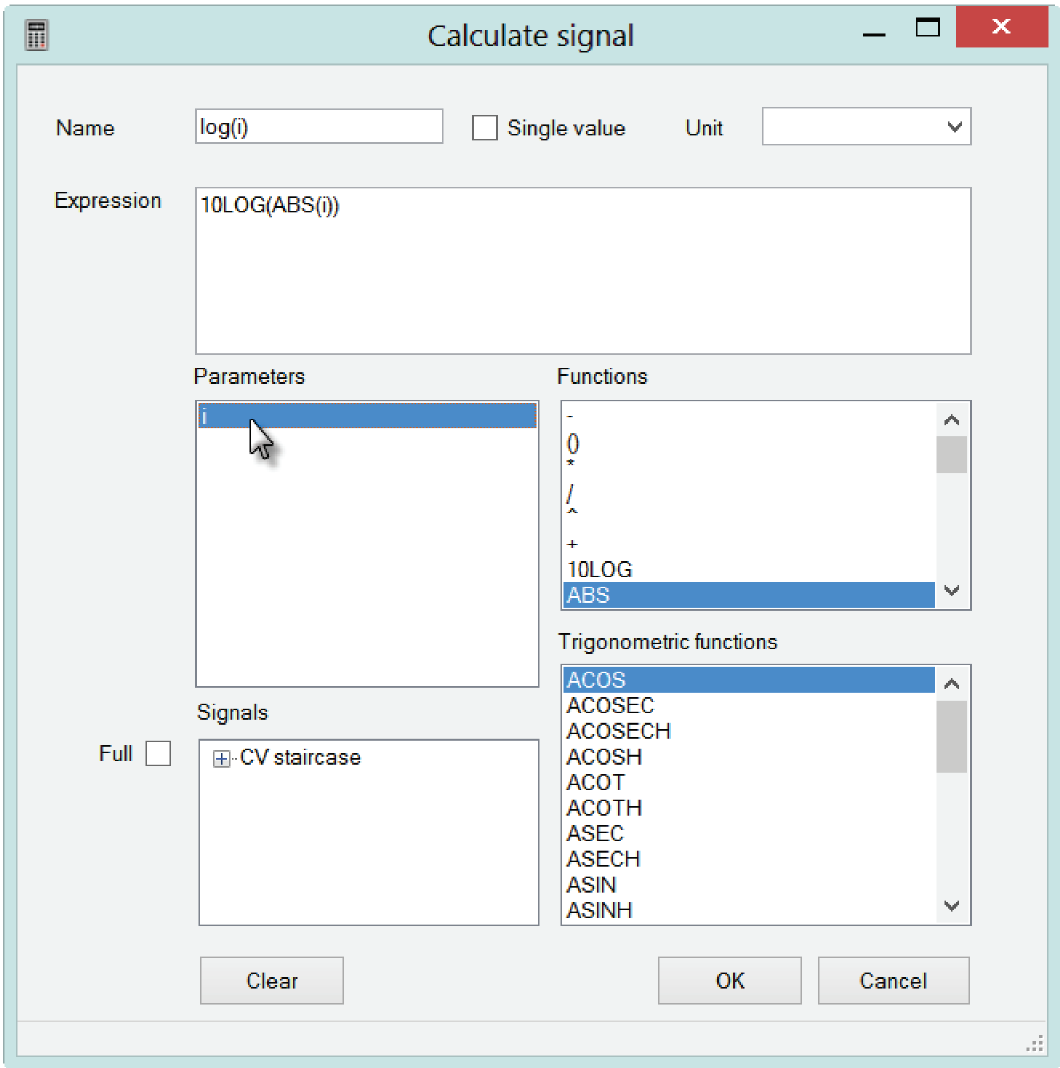


Figure 2.32 – Creating the log(i) signal – part 2: Identifying the parameters of the expression

The final step in the expression building process consists in linking the parameter(s) of the expression to existing signal(s). Expand the CV staircase list of available signals and **double click** the WE(1).Current signal to link it to the parameter **i** (see Figure 2.33).

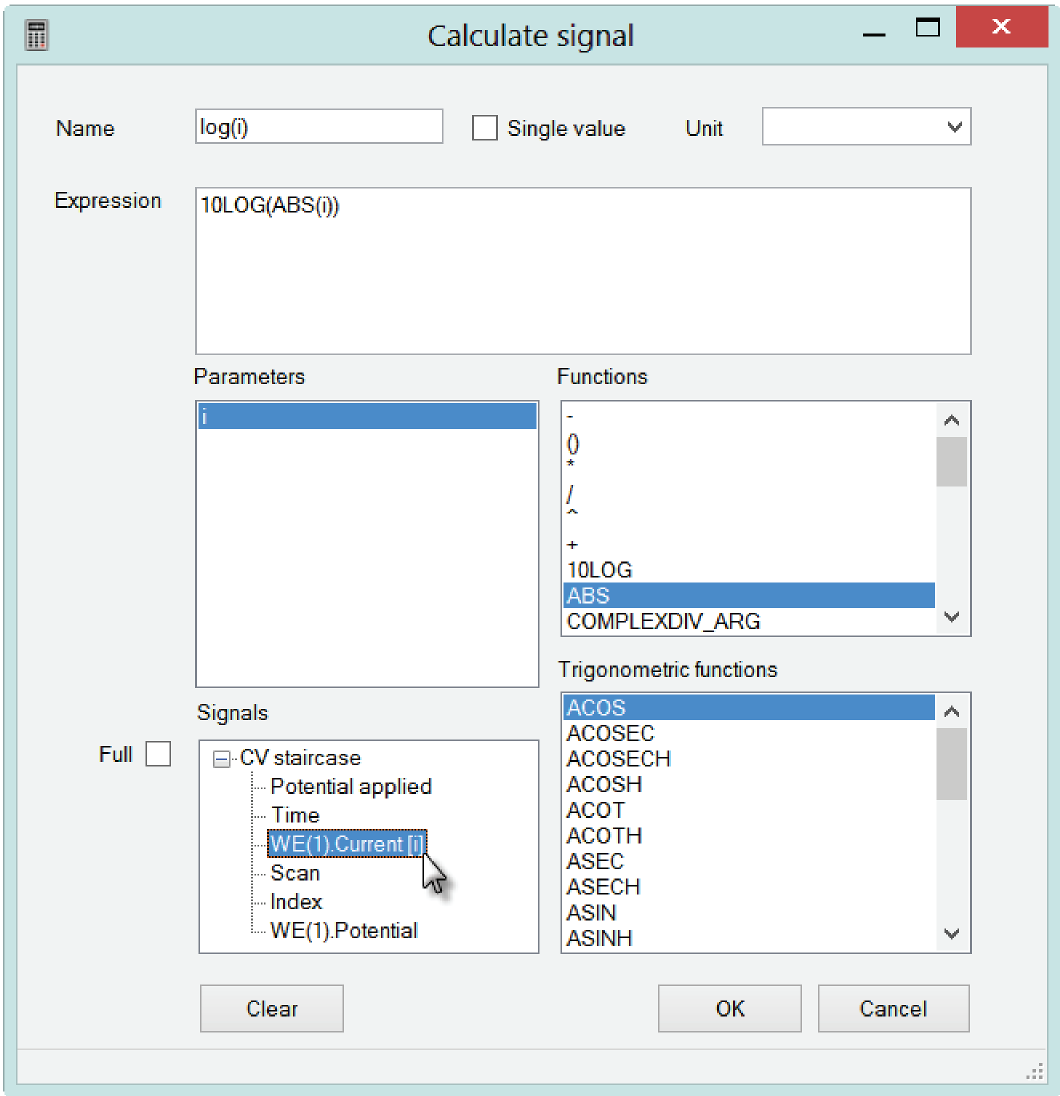


Figure 2.33 – Creating the log(i) signal – part 3: Linking the parameters of the expression to the available signals

The linked parameter will be displayed between brackets next to the name of the signal. The name of the signal will be displayed in red, indicating that it is linked to a parameter (see Figure 2.34).

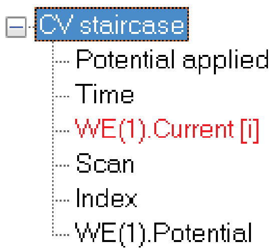


Figure 2.34 – A detailed view of the expression builder

Click the OK button to finish the calculation of the new signal. The contents of the data grid will be updated indicating that the new signal has been added to the list of available signals (see Figure 2.35). The expression used to calculate this signal is displayed in the calculation frame.

	Potential applied (V)	WE(1).Potential (V)	Time (s)	WE(1).Current (A)	Scan	Index	log(i)
▸	0	0.000839233	6.67146	1.15234E-6	1	1	-5.93842
	0.00244141	0.00322266	6.69586	3.38867E-6	1	2	-5.46997
	0.00488281	0.0056488	6.72026	5.61859E-6	1	3	-5.25037
	0.00732422	0.00805969	6.74466	7.84027E-6	1	4	-5.10567
	0.00976563	0.0105377	6.76906	1.00922E-5	1	5	-4.99602
	0.012207	0.0129852	6.79346	1.23352E-5	1	6	-4.90885
	0.0146484	0.0154297	6.81786	1.4566E-5	1	7	-4.83666
	0.0170898	0.0178619	6.84226	1.6806E-5	1	8	-4.77453
	0.0195313	0.0203308	6.86666	1.9046E-5	1	9	-4.7202
	0.0219727	0.0227722	6.89106	2.12921E-5	1	10	-4.67178

Figure 2.35 – The log(i) signal added to the data grid

The newly created log(i) signal can now be used as any other signal to plot the data either in 2D or 3D. Switch to the 2D plot by clicking the button in the toolbar. Set the plot settings for the X-axis to WE(1).Potential applied and for the Y-axis to **log(i)** as shown in Figure 2.36.

Note

The log(i) has been added to the list of signals available using the right click menu.

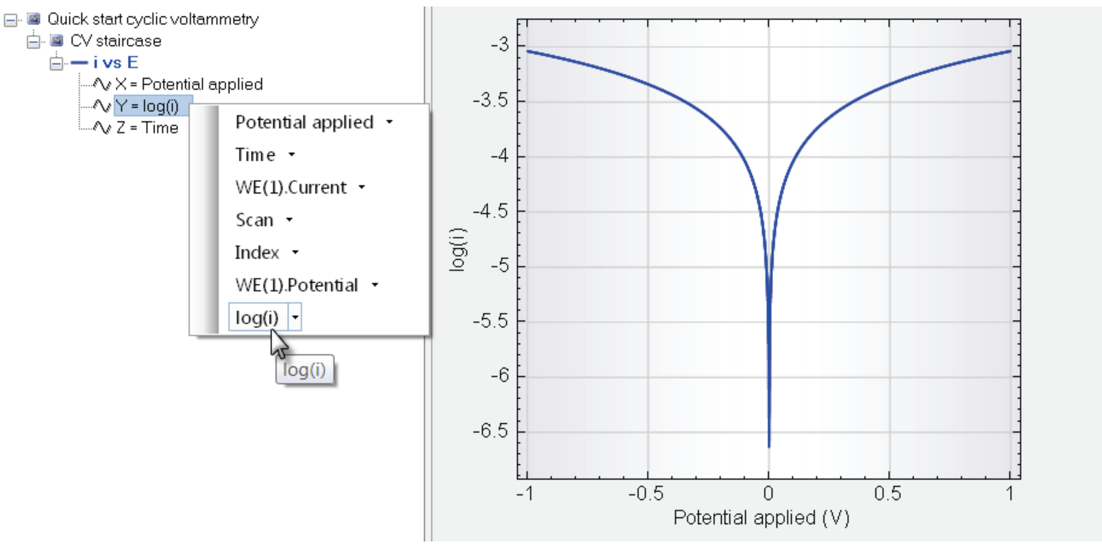


Figure 2.36 – Changing the plot settings to create the Tafel plot

2.2.5 – Saving to the database

In Nova, it is possible to save the changes in the database at any time. This allows you to keep all the modifications on a given data set, as well as the results of data analysis tools or additions to the data. To update a database entry, click the located in the analysis toolbar (see Figure 2.37).

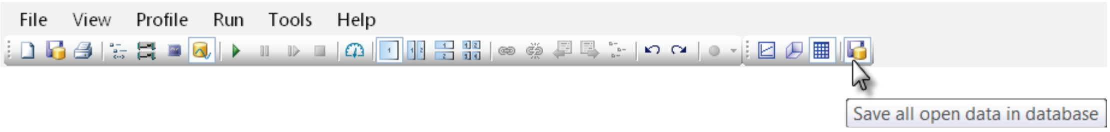


Figure 2.37 – Saving the modifications in the data base

Note

Saving the changes to the database in this case adds the log(i) signal to the data set as well as the plot settings (Tafel plot).

Note

We advise to go through the User Manual, chapter by chapter, since it provides in-depth information on procedures setup, measurements and data analysis.

Alternatively, you could skip to chapter 4 of the User Manual, which explores the Data analysis features of Nova in detail to further practice on the dummy cell data obtained in the course of this quick start.

Autolab procedures

Nova comes with a series of factory standard procedures, located in the Autolab group, that are available to every user and are intended both as examples and as simple measurement procedures.

This chapter provides an overview of the available factory standard procedures.

3 – The Autolab procedures group

The Autolab procedures group, located in the procedure browser frame contains a series of factory standard procedures. These procedures are intended to perform simple measurements and can be used for routine experiments or as templates for more elaborate procedures. The current version of Nova provides 26 Factory standard procedures (see Figure 3.1).

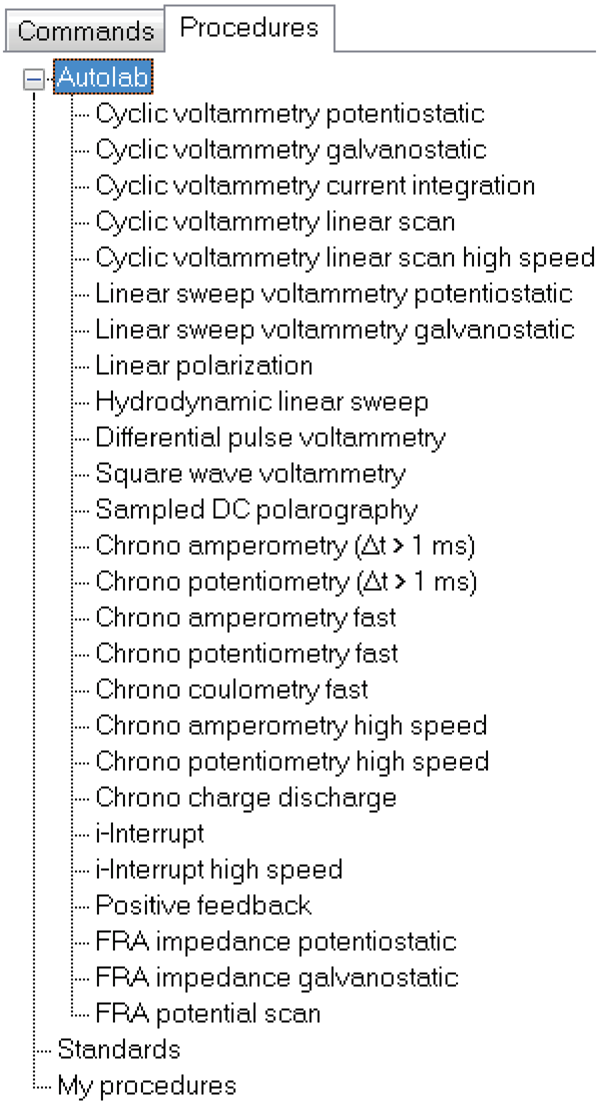


Figure 3.1 – The Autolab procedures group in the Procedure browser frame

The available procedures are:

- Cyclic voltammetry potentiostatic
- Cyclic voltammetry galvanostatic
- Cyclic voltammetry current integration[23]
- Cyclic voltammetry linear scan[24]
- Cyclic voltammetry linear scan high speed[24, 25]
- Linear sweep voltammetry potentiostatic
- Linear sweep voltammetry galvanostatic
- Linear polarization
- Hydrodynamic linear sweep[26]
- Differential pulse voltammetry[27]
- Square wave voltammetry[27]
- Sampled DC polarography[27]
- Chrono amperometry (Δt > 1 ms)
- Chrono potentiometry (Δt > 1 ms)
- Chrono amperometry fast
- Chrono potentiometry fast
- Chrono coulometry fast[23]
- Chrono amperometry high speed[25]
- Chrono potentiometry high speed[25]
- Chrono charge discharge
- i-Interrupt[28]
- i-Interrupt high speed[25, 28]
- Positive feedback[28]
- FRA impedance potentiostatic[29]
- FRA impedance galvanostatic[29]
- FRA potential scan[29]

Note

The actual number of procedures listed in the Autolab group of procedures in the Setup view depends on the active profile (Please refer to the User manual for more information on the use of profiles in Nova.). For example, if the Hardware based profile is active, only the procedures that are compatible with the connected Autolab are shown. Profiles can be selected from the Profile menu (see Figure 3.2).

[23] Requires the FI20 module or the on-board integrator (µAutolab II/III and PGSTAT101).
[24] Requires the SCANGEN or the SCAN250 module.
[25] Requires the ADC750 or the ADC10M.
[26] This procedure is intended to be used in combination with the Autolab RDE, using the Remote control option on the Autolab motor controller.
[27] The IME663 or the IME303 module must be declared in the Hardware setup.
[28] Not available on the µAutolab II/III and PGSTAT10.
[29] Requires the FRA2 or FRA32M module.

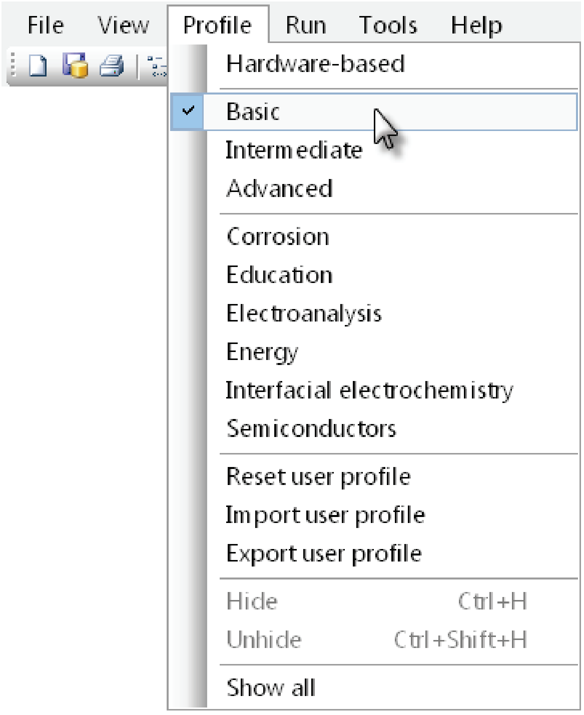


Figure 3.2 – The profile menu can be used to set one or more profiles active

This section provides information on the Autolab procedures. Some of these procedures are illustrated using the Autolab dummy cell. Procedures requiring additional hardware are not detailed. More information regarding the use of the optional modules is provided in the dedicated tutorials, available from the Help – Tutorials menu.

Each sub-section provides information on a specific procedure. A table is provided for each procedure listing the profile tags and hardware requirements (see Table 3.1).

Hardware Tags	None
Profile Tags	Basic
Application Tags	Energy

Table 3.1 – Table used to indicate the tags for an Autolab procedure (Cyclic voltammetry galvanostatic)

Table 3.1 shows the tags for the Autolab Cyclic voltammetry galvanostatic procedure. This procedure normally appears in the Basic profile and is also used in the Energy application profile. Furthermore, this procedure does not require additional hardware.

3.1 – Cyclic voltammetry potentiostatic

Hardware Tags	None
Profile Tags	Basic
Application Tags	Corrosion, Education, Electroanalysis, Energy, Interfacial electrochemistry, Semiconductors

The standard Cyclic voltammetry potentiostatic procedure is the first procedure located in the Autolab group of procedures. It is a typical potentiostatic staircase cyclic voltammetry procedure. The procedure has the following parameters:

- Preconditioning potential: 0 V
- Duration: 5 s
- CV Staircase:
  - Start potential: 0 V
  - Upper vertex potential: 1 V
  - Lower vertex potential: -1 V
  - Stop potential: 0 V
  - Number of stop crossings: 2
  - Step potential: 2.44 mV
  - Scan rate: 100 mV/s

Figure 3.3 shows an overview of the Cyclic voltammetry potentiostatic procedure.

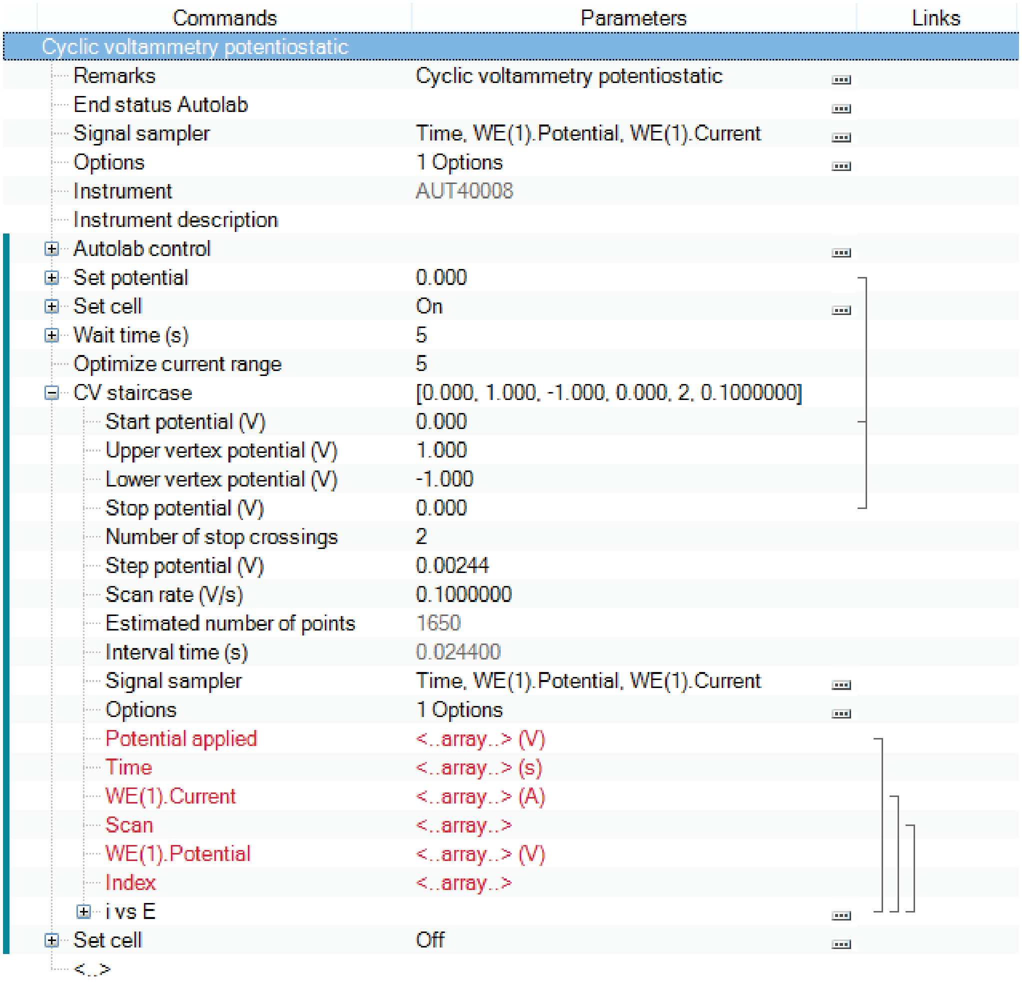


Figure 3.3 – The Cyclic voltammetry potentiostatic procedure

The signals sampled during this procedure are:

- Potential applied
- Time
- WE(1).Current
- Scan
- WE(1).Potential
- Index

The procedure uses the Automatic current ranging option and displays the measured data as WE(1).Current vs Potential applied in the measurement view. Figure 3.4 shows a measurement on the dummy cell (a) with the Autolab Cyclic voltammetry potentiostatic procedure.

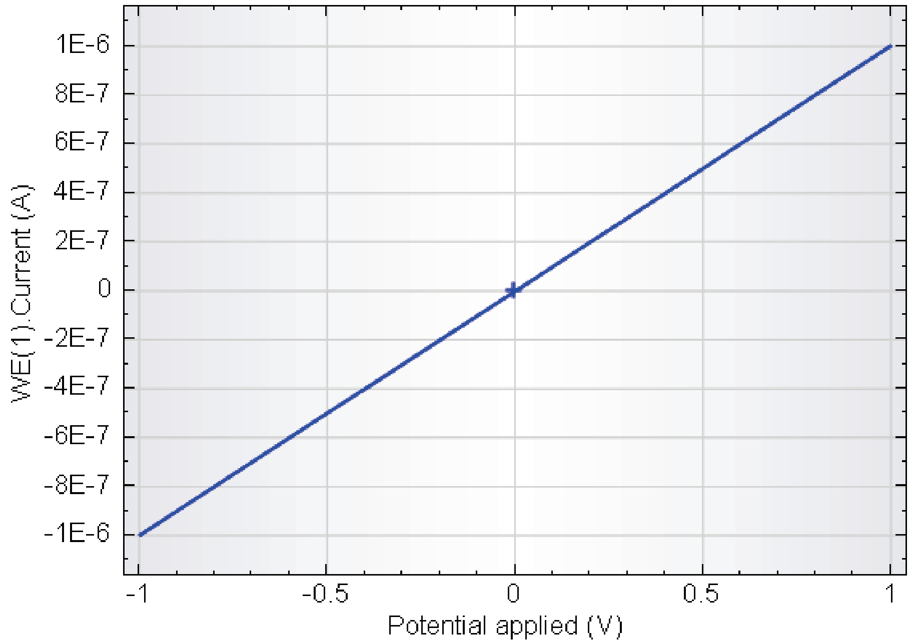


Figure 3.4 – The measured data obtained with the standard dummy cell (a) with the Cyclic voltammetry potentiostatic procedure

3.2 – Cyclic voltammetry galvanostatic

Hardware Tags	None
Profile Tags	Basic
Application Tags	Energy

The Cyclic voltammetry galvanostatic procedure is similar to the potentiostatic version. It is a typical galvanostatic staircase cyclic voltammetry procedure. The procedure has the following parameters:

- Preconditioning current: 0 A
- Duration: 5 s
- CV Staircase:
    - Start current: 0 A
    - Upper vertex current: 1 mA
    - Lower vertex current: -1 mA
    - Stop current: 0 V
    - Number of stop crossings: 2
    - Step current: 2.44 µA
    - Scan rate: 100 µA/s

Figure 3.5 shows an overview of the Cyclic voltammetry galvanostatic procedure.

Commands	Parameters	Links
Cyclic voltammetry galvanostatic		
Remarks	Cyclic voltammetry galvanostatic	
End status Autolab		
Signal sampler	Time, WE(1).Current, WE(1).Potential	
Options	No Options	
Instrument	AUT40008	
Instrument description		
Autolab control		
Set current	0.000E+00	
Set cell	On	
Wait time (s)	5	
CV staircase galvanostatic	[0.000E+00, 1.000E-03, -1.000E-03, 0.000E...	
Start current (A)	0.000E+00	
Upper vertex current (A)	1.000E-03	
Lower vertex current (A)	-1.000E-03	
Stop current (A)	0.000E+00	
Number of stop crossings	2	
Step current (A)	2.440E-06	
Scan rate (A/s)	1.000E-04	
Estimated number of points	1650	
Interval time (s)	0.024400	
Signal sampler	Time, WE(1).Current, WE(1).Potential	
Options	No Options	
Current applied	<..array..> (A)	
Time	<..array..> (s)	
Scan	<..array..>	
WE(1).Potential	<..array..> (V)	
WE(1).Current	<..array..> (A)	
Index	<..array..>	
E vs i		
Set cell	Off	
<..>		

Figure 3.5 – The Cyclic voltammetry galvanostatic procedure

The signals sampled during this procedure are:

- Current applied
- Time
- Scan
- WE(1).Potential
- WE(1).Current
- Index

Note

The automatic current ranging option is not available in galvanostatic mode (Please refer to Chapter 4 of this manual for more information on the Galvanostatic control restrictions.). This procedure uses the Autolab control command to set the instrument to galvanostatic mode and in the 1 mA current range before the measurement starts.

Figure 3.6 shows a measurement on the dummy cell (c) with the Autolab Cyclic voltammetry galvanostatic procedure.

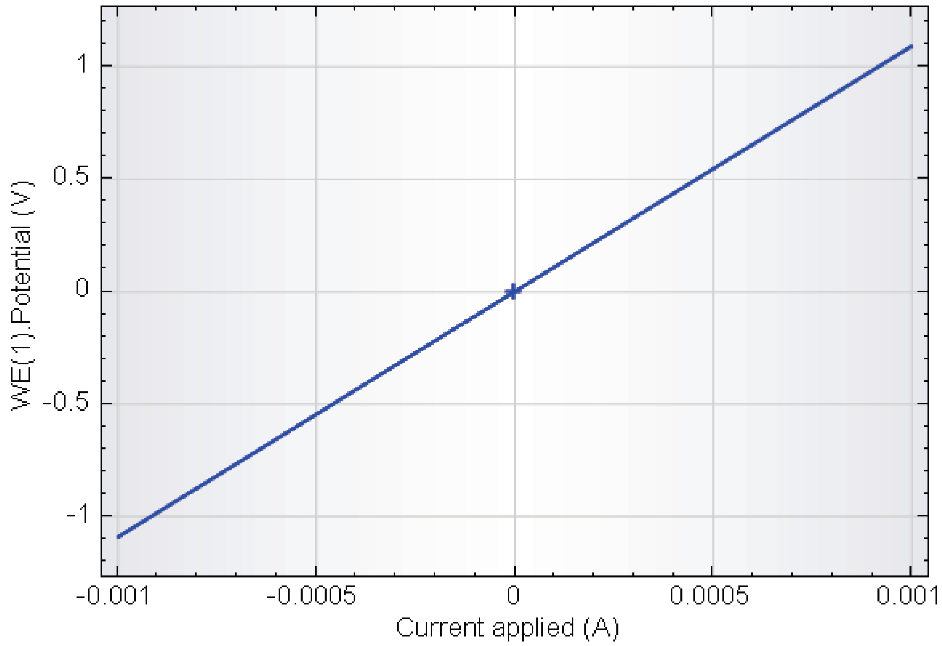


Figure 3.6 – The measured data obtained with the standard dummy cell (c) with the Cyclic voltammetry galvanostatic procedure

3.3 – Cyclic voltammetry current integration

Hardware Tags	FI20 or on-board integrator
Profile Tags	Basic
Application Tags	Education, Energy, Interfacial electrochemistry

This procedure requires the optional FI20 module or the on-board integrator for the µAutolabII/III and the PGSTAT101. The procedure can be used to perform a cyclic voltammogram using the current integration method. This measurement technique uses a staircase potential profile but rather than sampling the current at the end of each step to minimize capacitive currents, the total current is accumulated in the analog integrator.

At the end of each step, the accumulated charge is reconverted in current. This integrated current includes both the Faradaic and the capacitive currents passed during the potential step. If the interval time is large (typically > 20 ms), the current response measured during a current integration cyclic voltammetry experiment can be compared, in first approximation, to the current measured with a true linear scan potential profile. More information about the use of the analog integrator is provided in the **Filter and Integrator tutorial**, available from the Help menu in NOVA.

3.4 – Cyclic voltammetry linear scan

Hardware Tags	SCAN250 or SCANGEN module
Profile Tags	Basic
Application Tags	Education, Energy, Interfacial electrochemistry

This procedure requires the optional SCAN250 or SCANGEN module. Both modules are linear scan generators. The procedure can be used to perform a cyclic voltammogram using a true linear scan potential profile rather than a staircase potential profile. More information about the use of these modules is provided in the **Cyclic voltammetry linear scan tutorial**, available from the Help menu in NOVA.

3.5 – Cyclic voltammetry linear scan high speed

Hardware Tags	SCAN250 or SCANGEN in combination with ADC10M or ADC750 module
Profile Tags	Basic
Application Tags	None

This procedure requires the optional SCAN250 or SCANGEN module and the optional ADC10M or ADC750 module. The SCAN250 and the SCANGEN are both linear scan generators. The ADC10M and the ADC750 are fast sampling analog to digital converters. The procedure can be used to perform a cyclic voltammogram using a true linear scan potential profile rather than a staircase potential profile, at high scan rate[30]. More information about the use of these modules is provided in the **Cyclic voltammetry linear scan tutorial**, available from the Help menu in NOVA.

[30] Up to 10 kV/s with the SCANGEN+ADC750 or ADC10M and the SCAN250+ADC750; up to 250 kV/s with the SCAN250 + ADC10M.

3.6 – Linear sweep voltammetry potentiostatic

Hardware Tags	None
Profile Tags	Basic
Application Tags	Corrosion, Education, Electroanalysis, Energy, Interfacial electrochemistry, Semiconductors

This procedure is a typical example of a staircase linear sweep voltammetry experiment in potentiostatic conditions. The procedure has the following parameters:

- Preconditioning potential: 0 V
- Duration: 5 s
- CV Staircase:
    - Start potential: 0 V
    - Stop potential: 1 V
    - Step potential: 2.44 mV
    - Scan rate: 100 mV/s

Figure 3.7 shows an overview of the Linear sweep voltammetry potentiostatic procedure.

Commands	Parameters	Links
Linear sweep voltammetry potentiostatic		
Remarks	Linear sweep voltammetry potentiostatic	
End status Autolab		
Signal sampler	Time, WE(1).Potential, WE(1).Current	
Options	1 Options	
Instrument	AUT40008	
Instrument description		
Autolab control		
Set potential	0.000	
Set cell	On	
Wait time (s)	5	
Optimize current range	5	
LSV staircase	[0.000, 1.000, 0.1000000]	
Start potential (V)	0.000	
Stop potential (V)	1.000	
Step potential (V)	0.00244	
Scan rate (V/s)	0.1000000	
Estimated number of points	422	
Interval time (s)	0.024400	
Signal sampler	Time, WE(1).Potential, WE(1).Current	
Options	1 Options	
Potential applied	<..array..> (V)	
Time	<..array..> (s)	
WE(1).Current	<..array..> (A)	
WE(1).Potential	<..array..> (V)	
Index	<..array..>	
i vs E		
Set cell	Off	
<..>		

Figure 3.7 – The Linear sweep voltammetry potentiostatic procedure

The signals sampled during this procedure are:

- Potential applied
- Time
- WE(1).Current
- WE(1).Potential
- Index

The procedure uses the Automatic current ranging option and displays the measured data as WE(1).Current vs Potential applied in the measurement view. Figure 3.8 shows a measurement on the dummy cell (a) with the Autolab Linear sweep voltammetry potentiostatic procedure.

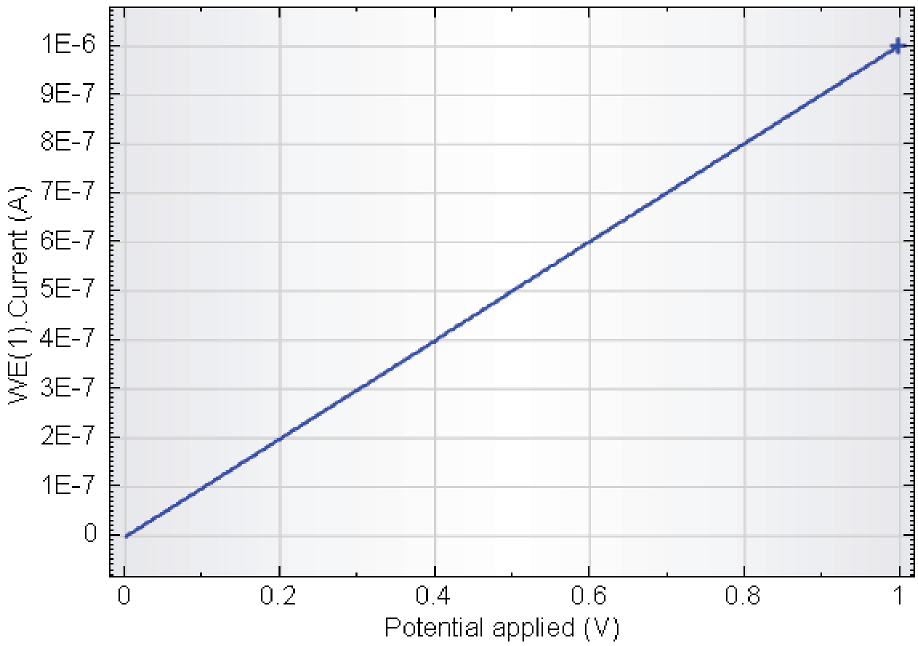


Figure 3.8 – The measured data obtained with the standard dummy cell (a) with the Linear sweep voltammetry potentiostatic procedure

3.7 – Linear sweep voltammetry galvanostatic

Hardware Tags	None
Profile Tags	Basic
Application Tags	Energy

This procedure is a typical example of a staircase linear sweep voltammetry experiment in galvanostatic conditions. The procedure has the following parameters:

- Preconditioning current: 0 A
- Duration: 5 s
- CV Staircase:
    - Start current: 0 A
    - Stop current: 1 mA
    - Step current: 2.44 μA
    - Scan rate: 100 μA/s

Figure 3.9 shows an overview of the Linear sweep voltammetry galvanostatic procedure.

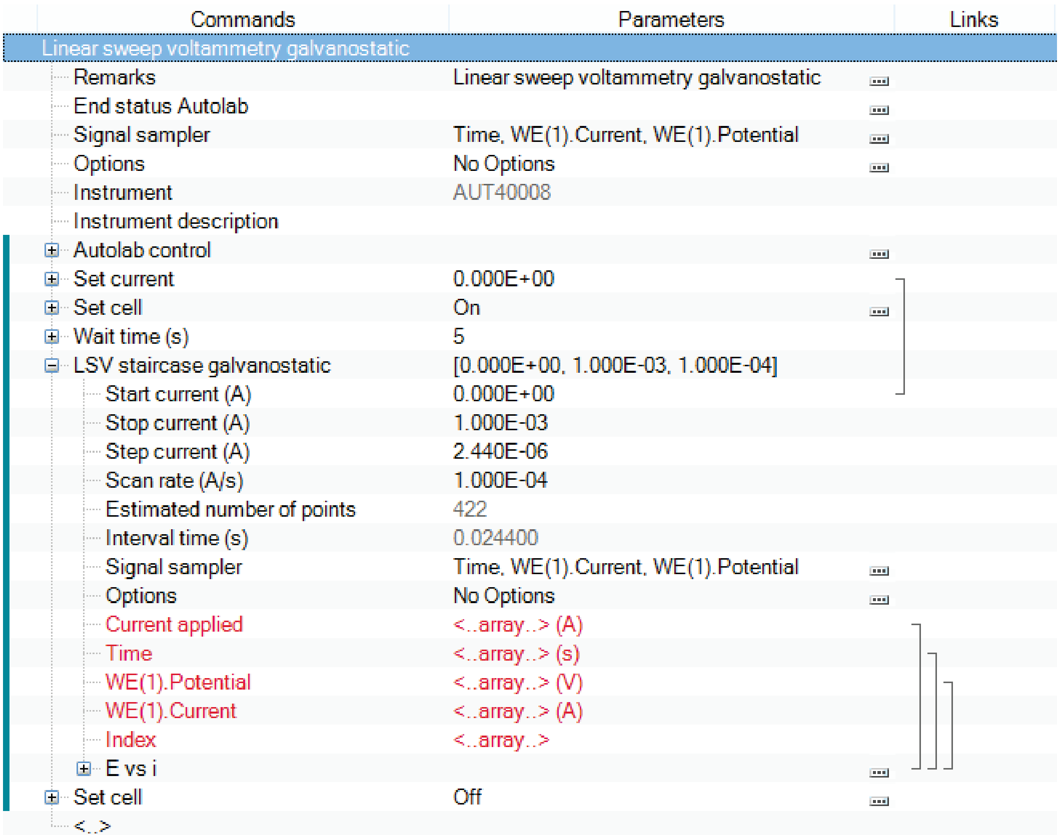


Figure 3.9 – The Linear sweep voltammetry galvanostatic procedure

The signals sampled during this procedure are:

- Current applied
- Time
- WE(1).Potential
- WE(1).Current
- Index

Note

The automatic current ranging option is not available in galvanostatic mode (Please refer to Chapter 4 of this manual for more information on the Galvanostatic control restrictions.). This procedure uses the Autolab control command to set the instrument to galvanostatic mode and in the 1 mA current range before the measurement starts.

Figure 3.10 shows a measurement on the dummy cell (c) with the Autolab Linear sweep voltammetry galvanostatic procedure.

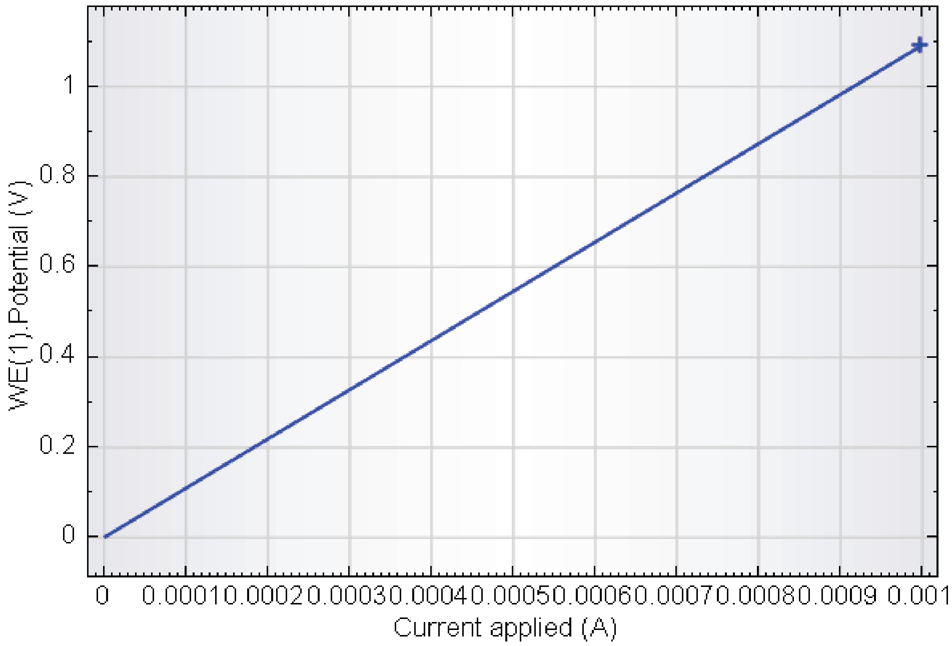


Figure 3.10 – The measured data obtained with the standard dummy cell (c) with the Linear sweep voltammetry galvanostatic procedure

3.8 – Linear polarization

Hardware Tags	None
Profile Tags	Basic
Application Tags	Corrosion

The Linear polarization procedure measures the OCP potential (using the *OCP determination* command[31]) for the sample and then uses the *Set reference potential* command set the potential values of the linear sweep voltammetry relative to the averaged OCP (a moving average of 5 seconds is used).

The Linear polarization procedure has the following parameters:

- Measure OCP for 120 seconds with cutoff when dOCP/dt < 1 µV/s
- Preconditioning potential: -100 mV (vs. OCP)
- Duration: 5 s
- LSV Staircase:
    - Start potential: -100 mV (vs. OCP)
    - Stop potential: 100 mV (vs. OCP)
    - Step potential: 1 mV
    - Scan rate: 1 mV/s

Figure 3.11 shows an overview of the Linear polarization procedure.

[31] Please refer to the Open circuit potential Tutorial, available from the Help menu, for more information on the OCP determination command.

Commands	Parameters	Links
Linear polarization		
Remarks	Linear polarization	
End status Autolab		
Signal sampler	Time, WE(1).Potential, WE(1).Current	
Options	1 Options	
Instrument	AUT40008	
Instrument description		
Autolab control		
OCP determination	[0.000]	
Set reference potential	0.000	
Set potential	-0.100	
Set cell	On	
Wait time (s)	5	
Optimize current range	5	
LSV staircase	[-0.100, 0.100, 0.0010000]	
Start potential (V)	-0.100	
Stop potential (V)	0.100	
Step potential (V)	0.00100	
Scan rate (V/s)	0.0010000	
Estimated number of points	200	
Interval time (s)	1.000000	
Signal sampler	Time, WE(1).Potential, WE(1).Current	
Options	1 Options	
Potential applied	<..array..> (V)	
Time	<..array..> (s)	
WE(1).Current	<..array..> (A)	
WE(1).Potential	<..array..> (V)	
Index	<..array..>	
Log(i) vs E		
Corrosion rate, fit		
Set cell	Off	
<..>		

Figure 3.11 – The standard Linear polarization procedure

During the OCP determination, the following signals are sampled:

- Time
- WE(1).Potential

The signals sampled during the linear sweep voltammetry measurement are:

- Potential applied
- Time
- WE(1).Current
- WE(1).Potential
- Index

At the end of the measurement, a corrosion rate calculation is performed.

Figure 3.12 shows a measurement on the dummy cell (c) with the Autolab Linear polarization procedure.

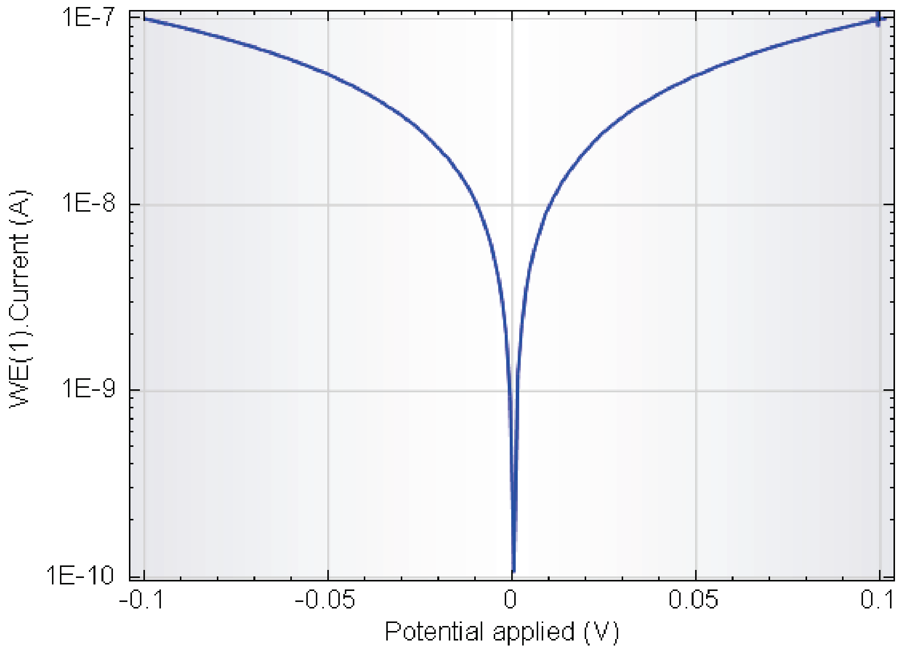


Figure 3.12 – The measured data obtained with the standard dummy cell (a) with the Linear polarization procedure

3.9 – Hydrodynamic linear sweep

Hardware Tags	Autolab RDE connected to DAC164 or V_{out} of Autolab
Profile Tags	Basic
Application Tags	Corrosion, Education, Electroanalysis, Energy, Interfacial electrochemistry

The Hydrodynamic linear sweep voltammetry procedure performs a linear sweep voltammetry using the Autolab RDE, with six different rotation rates. The rotation rate of the Autolab RDE is set using the *Control Autolab RDE* command linked to the values of a *repeat for each value* command[32].

This procedure is intended to be used with the Remote switch of the Autolab motor controller enabled (on the back plane of the controller) and with a BNC cable connected between the DAC164 ←1 connector (V_{out} for the µAutolab II, III, the PGSTAT101, PGSTAT204 and the Multi Autolab) and the Remote input plug on the back plane of the Autolab RDE motor controller (see Figure 3.13).

[32] Remote control of the Autolab RDE requires a BNC cable between the Autolab and the Autolab motor controller.

Figure 3.13 – The Hydrodynamic linear sweep Voltammetry is intended to be used with the Autolab RDE and motor controller

Please refer to the Autolab RDE User Manual for more information.

The Hydrodynamic linear sweep voltammetry has the following parameters:

- Preconditioning potential: 1 V
- Set RDE rotation rate to 0 RPM
- Duration: 15 s
- Repeat for each value
    - Set RDE rotation rate
    - Wait 15 s
    - LSV Staircase
        - Start potential: 1 V
        - Stop potential: 0 V
        - Step potential: -2.44 mV
        - Scan rate: 100 mV/s

Figure 3.14 shows an overview of the Hydrodynamic linear sweep voltammetry procedure.

Commands	Parameters	Links
Hydrodynamic linear sweep		
Remarks	Hydrodynamic linear sweep: requires an RDE connected	
End status Autolab		
Signal sampler	Time, WE(1).Potential, WE(1).Current	
Options	1 Options	
Instrument	AUT40008	
Instrument description		
Control Autolab RDE	0	
Autolab control		
Set potential	1.000	
Set cell	On	
Wait time (s)	15	
Repeat for each value	500; 831.92; 1247.9; 1747.9; 2331.9; 3000	
Number of repetitions	6	
Parameter link	500	
Set potential	1.000	
Control Autolab RDE	500	
DAC channel	3	
Rotation rate	500	
ω	<..array..> (rad/s)	
Calculate ω	2.0*PI*RPM/60.0	
Wait time (s)	15	
Optimize current range	5	
LSV staircase	[1.000, 0.000, 0.1000000]	
Start potential (V)	1.000	
Stop potential (V)	0.000	
Step potential (V)	-0.00244	
Scan rate (V/s)	0.1000000	
Estimated number of points	422	
Interval time (s)	0.024400	
Signal sampler	Time, WE(1).Potential, WE(1).Current	
Options	1 Options	
Potential applied	<..array..> (V)	
Time	<..array..> (s)	
WE(1).Current	<..array..> (A)	
WE(1).Potential	<..array..> (V)	
Index	<..array..>	
i vs E		
<..>		
Switch Autolab RDE off		
Set cell	Off	
Hydrodynamic i vs √ω		
<..>		

Figure 3.14 – The standard Hydrodynamic linear sweep procedure

The signals sampled during this procedure are:

- Potential applied
- Time
- WE(1).Current
- WE(1).Potential
- Index

Note

The step value used in the Hydrodynamic linear sweep voltammetry procedure is negative because the sweep goes from 1 V to 0 V.

3.10 – Differential pulse voltammetry

Hardware Tags	IME663 or IME303 interface
Profile Tags	Basic
Application Tags	Electroanalysis

This procedure, intended to be used in combination with a Mercury Drop Electrode (MDE) stand (Metrohm 663 VA, Princeton Applied Research 303/303A or other compatible MDE) provides an example of a differential pulse voltammetry measurement in NOVA.

This procedure requires the optional IME663 or IME303 module to be selected in the Hardware setup. More information about the use of these Autolab accessories is provided in the **Voltammetric analysis tutorial**, available from the Help menu in NOVA.

3.11 – Square wave voltammetry

Hardware Tags	IME663 or IME303 interface
Profile Tags	Basic
Application Tags	Electroanalysis

This procedure, intended to be used in combination with a MDE stand (Metrohm 663 VA, Princeton Applied Research 303/303A or other compatible MDE) provides an example of a square wave voltammetry measurement in NOVA.

This procedure requires the optional IME663 or IME303 module to be selected in the Hardware setup. More information about the use of these Autolab accessories is provided in the **Voltammetric analysis tutorial**, available from the Help menu in NOVA.

3.12 – Sampled DC polarography

Hardware Tags	IME663 or IME303 interface
Profile Tags	Basic
Application Tags	Electroanalysis

This procedure, intended to be used in combination with a MDE stand (Metrohm 663 VA, Princeton Applied Research 303/303A or other compatible MDE) provides

an example of a sampled DC polarography measurement in NOVA. During this procedure, a new Hg drop is created at the end of each potential step.

This procedure requires the optional IME663 or IME303 module to be selected in the Hardware setup. More information about the use of these Autolab accessories is provided in the **Voltammetric analysis tutorial**, available from the Help menu in NOVA.

3.13 – Chrono amperometry (Δt > 1 ms)

Hardware Tags	None
Profile Tags	Basic
Application Tags	Corrosion, Education, Electroanalysis, Energy, Interfacial electrochemistry, Semiconductors

The Chrono amperometry (Δt > 1 ms) procedure has three consecutive potential steps. After each potential step, the current response is recorded during five seconds, with an interval time of 10 ms. The *Record signals (>1 ms)* command is used to measure the electrochemical signals. This command samples the signals with a smallest possible interval time of 1.30 ms.

The procedure has the following parameters:

- Preconditioning potential: 0 V
- Duration: 5 s
- Potential step 1: 0 V
- Potential step 2: 0.5 V
- Potential step 3: -0.5 V

Figure 3.15 shows an overview of the Chrono amperometry (Δt > 1 ms) procedure.

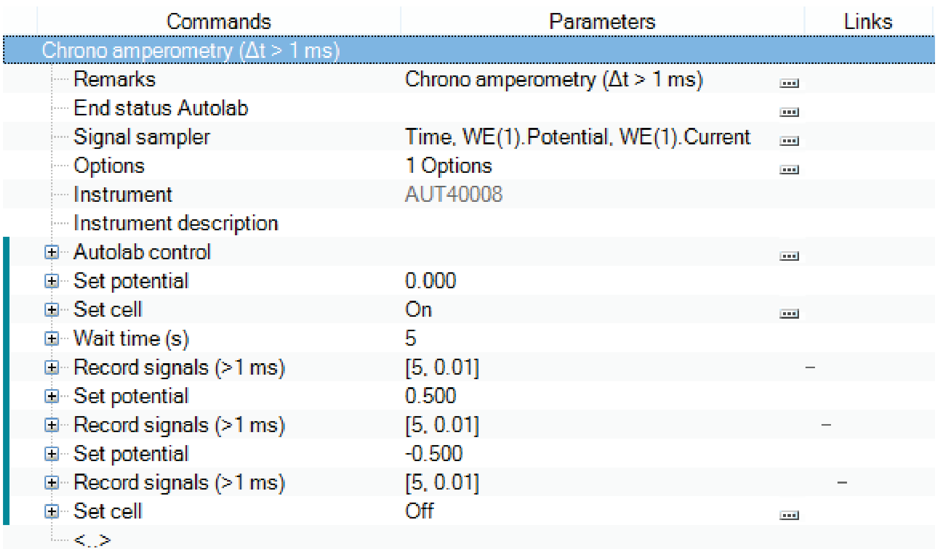


Figure 3.15 – The Chrono amperometry (Δt > 1 ms) procedure

The signals sampled during this procedure are:

- Corrected time
- WE(1).Potential
- WE(1).Current
- Time
- Index

Figure 3.16 shows a measurement on the dummy cell (a) with the Autolab Chrono amperometry (Δt > 1 ms) procedure.

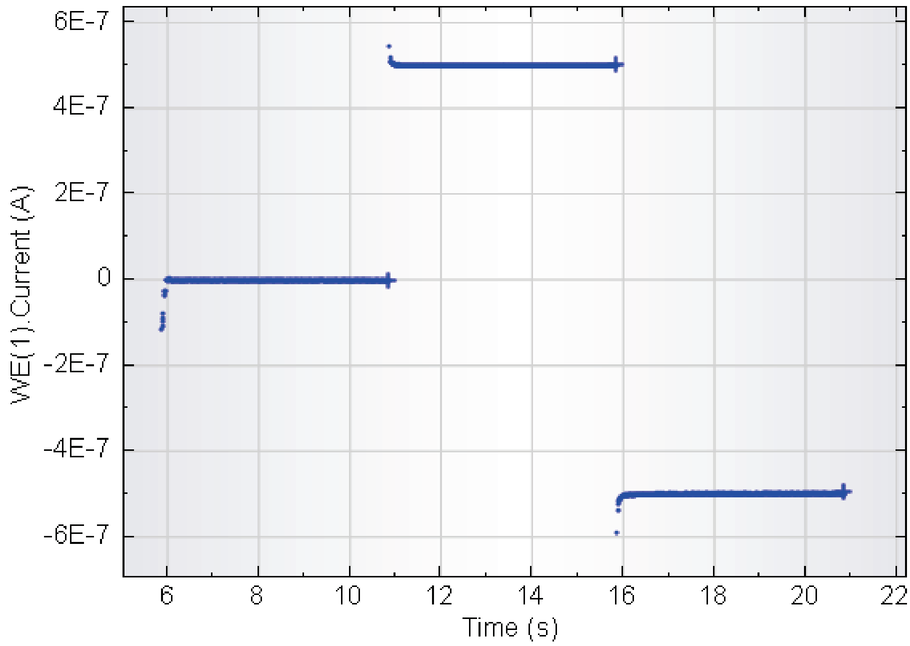


Figure 3.16 – The measured data obtained with the standard dummy cell (a) with the Chrono amperometry (Δt > 1 ms) procedure

3.14 – Chrono potentiometry (Δt > 1 ms)

Hardware Tags	None
Profile Tags	Basic
Application Tags	Corrosion, Energy, Interfacial electrochemistry

The Chrono potentiometry (Δt > 1 ms) procedure has three consecutive current steps. After each current step, the potential response is recorded during five seconds, with an interval time of 10 ms. The *Record signals (>1 ms)* command is used to measure the electrochemical signals. This command samples the signals with a smallest possible interval time of 1.30 ms.

The procedure has the following parameters:

- Preconditioning current: 0 A
- Duration: 5 s
- Potential step 1: 0 A
- Potential step 2: 0.5 mA
- Potential step 3: -0.5 mA

Figure 3.17 shows an overview of the Chrono potentiometry (Δt > 1 ms) procedure.

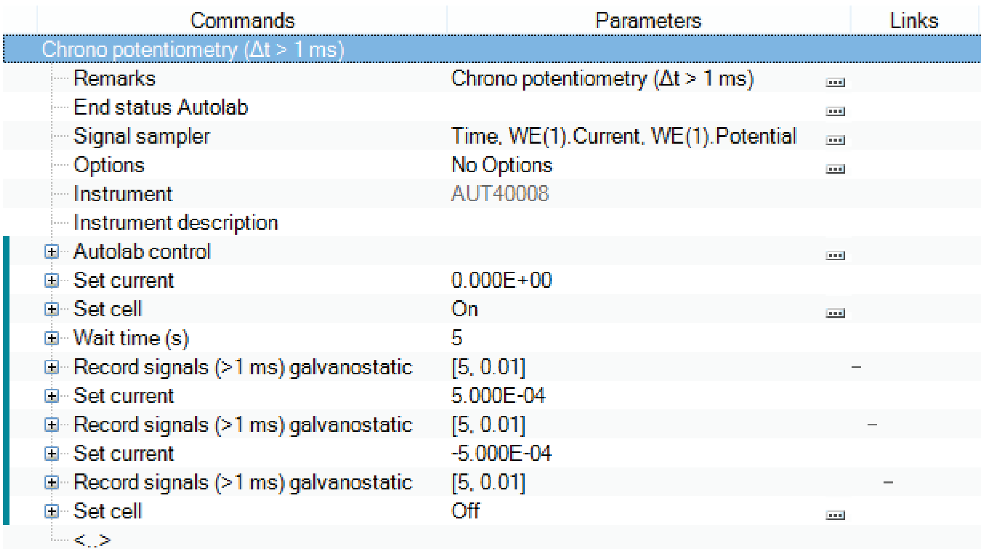


Figure 3.17 – The Chrono potentiometry (Δt > 1 ms) procedure

The signals sampled during this procedure are:

- Corrected time
- WE(1).Potential
- WE(1).Current
- Time
- Index

Figure 3.18 shows a measurement on the dummy cell (c) with the Autolab Chrono potentiometry (Δt > 1 ms) procedure.

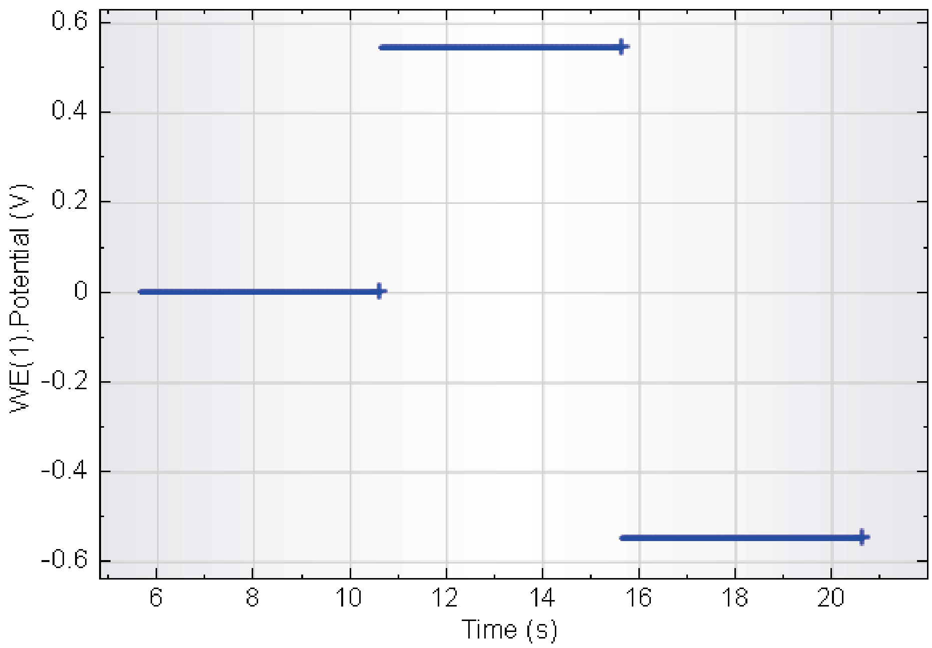


Figure 3.18 – The measured data obtained with the standard dummy cell (c) with the Chrono potentiometry (Δt > 1 ms) procedure

3.15 – Chrono amperometry fast

Hardware Tags	None
Profile Tags	Basic
Application Tags	Interfacial electrochemistry

The Chrono amperometry fast procedure uses the *Chrono methods* command instead of the *Record signals* command. The Chrono methods command can be used for fast electrochemical measurements. The interval time can be lower than 1 ms[33]. Because this command works with higher sampling rates compared to the *Record signals* command, the data cannot be plotted real-time. The measured data is displayed at the end of the measurement.

The procedure has the following parameters:

- Preconditioning potential: 0 V
- Duration: 5 s
- Potential step 1: 0 V
- Potential step 2: 0.3 V
- Potential step 3: -0.3 V
- Potential step 4: 0 V

[33] Down to ~ 100 µs.

The response of the cell is measured with an interval time of 100 µs. At the end of the measurement, switch to the analysis view to see the measured data points.

Figure 3.19 shows an overview of the Chrono amperometry fast procedure.

Commands	Parameters	Links
Chrono amperometry fast		
Remarks	Chrono amperometry fast	
End status Autolab		
Signal sampler	Time, WE(1).Current	
Options	No Options	
Instrument	AUT40008	
Instrument description		
Autolab control		
Set potential	0.000	
Set cell	On	
Wait time (s)	5	
Chrono methods	[1, .04]	
Number of repeats	1	
Total duration (s)	.04	
Estimated number of points	400	
Signal sampler	Time, WE(1).Current	
Corrected time	<..array..> (s)	
Level	<..array..>	
Time	<..array..> (s)	
WE(1).Current	<..array..> (A)	
Index	<..array..>	
i vs t		
Set cell	Off	
<..>		

Figure 3.19 – The Chrono amperometry fast procedure

The levels used in this procedure are shown in Figure 3.20.

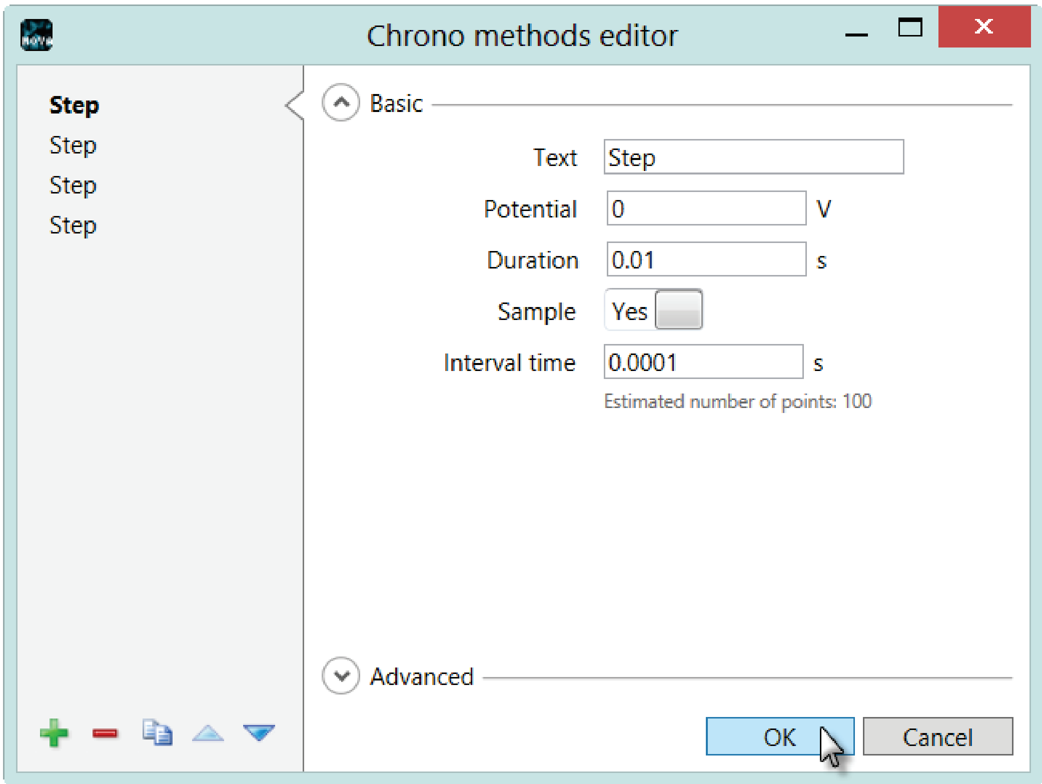


Figure 3.20 – Overview of the levels used in the Chrono amperometry fast procedure

The signals sampled during this procedure are:

- Corrected time
- Level
- Time
- WE(1).Current
- Index

Note

The automatic current ranging option is not available during the chrono methods measurement. This procedure uses the *Autolab control* command to set the instrument high speed and in the 1 mA current range before the measurement starts.

Figure 3.21 shows a measurement on the dummy cell (c) with the Autolab Chrono amperometry fast procedure.

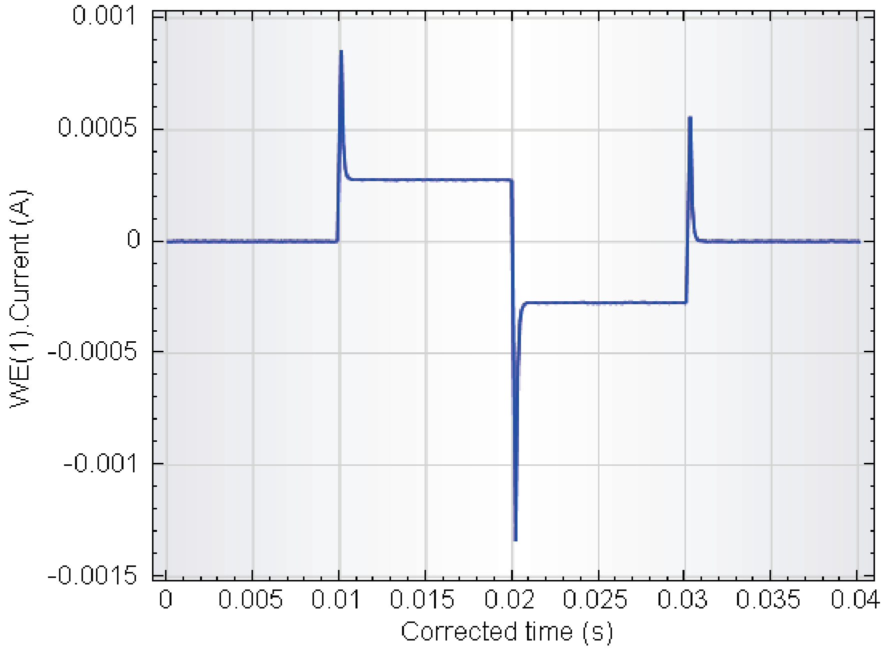


Figure 3.21 – The measured data obtained with the standard dummy cell (c) with the Chrono amperometry fast procedure

Note

More information on time resolved measurements can be found in the Chrono methods tutorial, available from the Help menu in NOVA.

3.16 – Chrono potentiometry fast

Hardware Tags	None
Profile Tags	Basic
Application Tags	Interfacial electrochemistry

The Chrono potentiometry fast procedure uses the *Chrono methods* command instead of the *Record signals* command. The Chrono methods command can be used for fast electrochemical measurements. The interval time can be lower than 1 ms[34]. Because this command works with higher sampling rates compared to the *Record signals* command, the data cannot be plotted real-time.

The procedure has the following parameters:

- Preconditioning current: 0 A
- Duration: 5 s
- Potential step 1: 0 A
- Potential step 2: 3 A
- Potential step 3: -3 A
- Potential step 4: 0 A

The response of the cell is measured with an interval time of 100 µs. At the end of the measurement, switch to the analysis view to see the measured data points.

Figure 3.22 shows an overview of the Chrono potentiometry fast procedure.

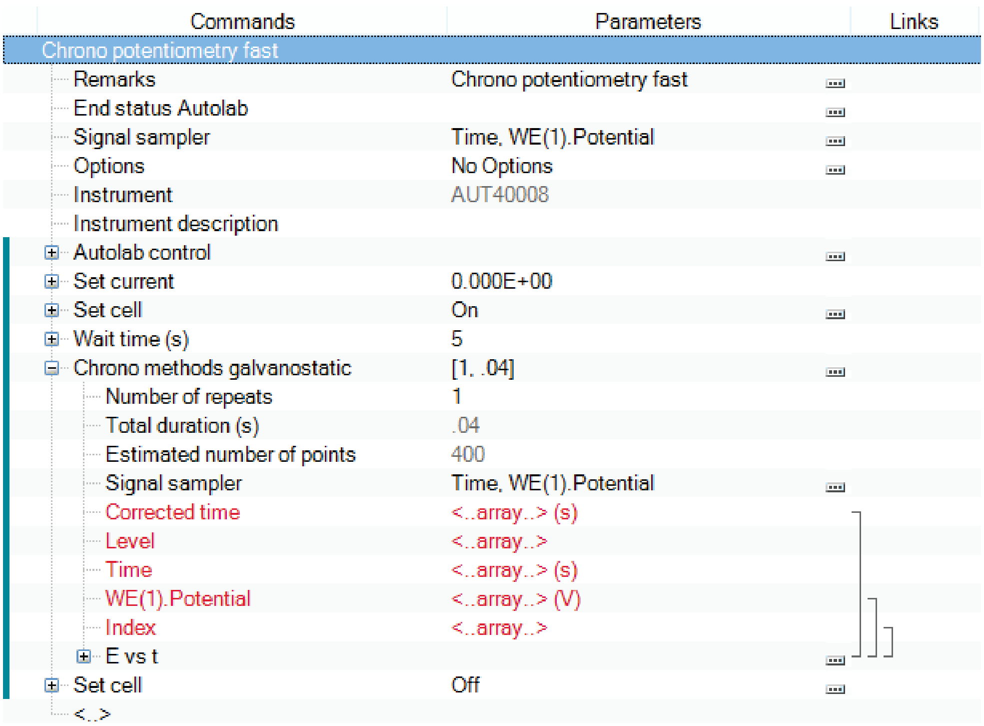


Figure 3.22 – The Chrono potentiometry fast procedure

[34] Down to ~ 100 µs.

The levels used in this procedure are shown in Figure 3.23.

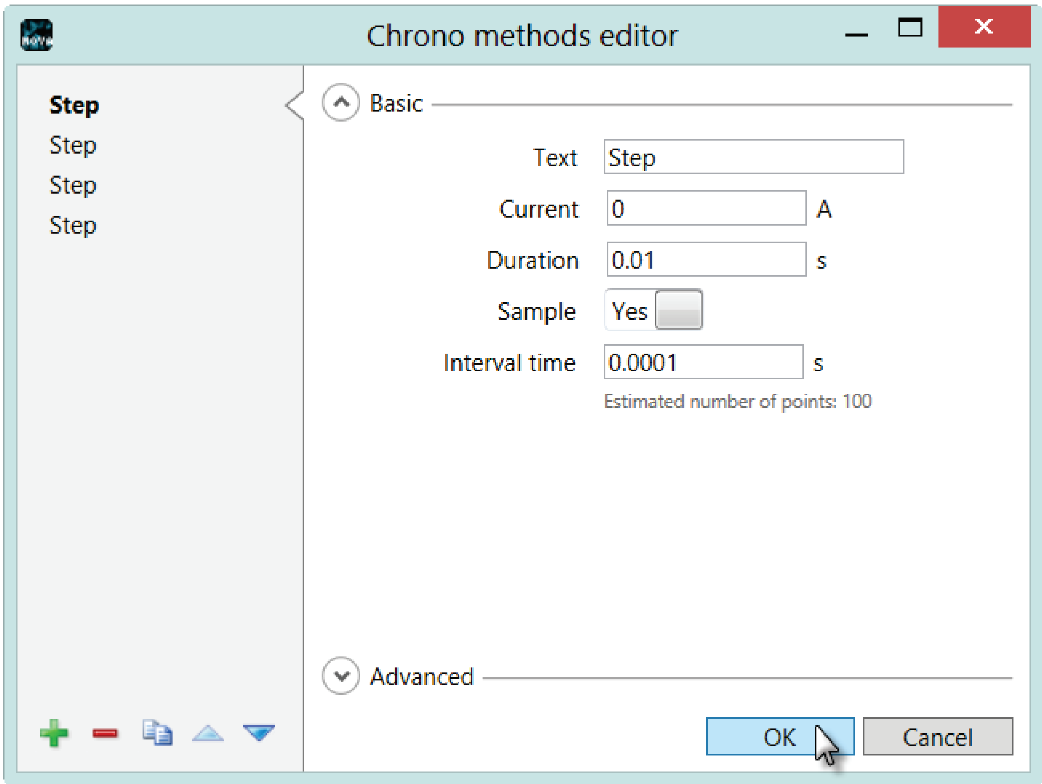


Figure 3.23 – Overview of the levels used in the Chrono potentiometry fast procedure

The signals sampled during this procedure are:

- Corrected time
- Level
- Time
- WE(1).Potential
- Index

Note

The automatic current ranging option is not available during the galvanostatic chrono methods measurement. This procedure uses the Autolab control command to set the instrument to galvanostatic mode, high speed and in the 1 mA current range before the measurement starts.

Figure 3.24 shows a measurement on the dummy cell (c) with the Autolab Chrono potentiometry fast procedure.

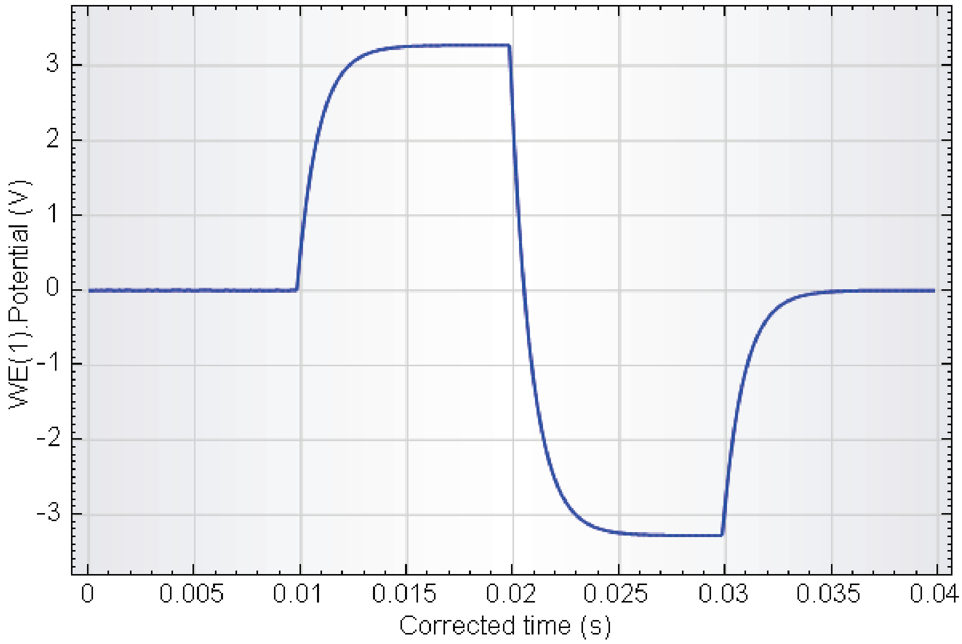


Figure 3.24 – The measured data obtained with the standard dummy cell (c) with the Chrono potentiometry fast procedure

3.17 – Chrono coulometry fast

Hardware Tags	FI20 or on-board integrator
Profile Tags	Basic
Application Tags	Interfacial electrochemistry

This procedure requires the optional FI20 module or the on-board integrator for the µAutolabII/III, the PGSTAT101 and Multi Autolab with M101. The procedure can be used to perform chrono coulometric measurements. The integrator module provides a direct measurement of the charge. More information about the use of the analog integrator is provided in the **Filter and Integrator tutorial**, available from the Help menu in NOVA.

3.18 – Chrono amperometry high speed

Hardware Tags	ADC10M or ADC750 module
Profile Tags	Basic
Application Tags	None

The Chrono amperometry high speed procedure uses the *Chrono methods high speed* command. This command requires the optional ADC10M or ADC750 module. Depending on the module, the shortest interval time is 100 ns (ADC10M) or 1.33 µs (ADC750).

More information about the use of these modules is provided in the **Chrono methods high speed tutorial**, available from the Help menu in NOVA.

3.19 – Chrono potentiometry high speed

Hardware Tags	ADC10M or ADC750
Profile Tags	Basic
Application Tags	None

The Chrono potentiometry high speed procedure uses the *Chrono methods high speed* command. This command requires the optional ADC10M or ADC750 module. Depending on the module, the shortest interval time is 100 ns (ADC10M) or 1.33 µs (ADC750).

More information about the use of these modules is provided in the **Chrono methods high speed tutorial**, available from the Help menu in NOVA.

3.20 – Chrono charge discharge

Hardware Tags	None
Profile Tags	Basic
Application Tags	Energy

The Chrono charge discharge procedure uses the *Repeat n times* command to repeat a combination of *Set potential* and *Record signals (>1 ms)* sequence. The response of the cell is recorded during 2.5 s, with an interval time of 10 ms. The Chrono charge discharge procedure has the following parameters:

- Preconditioning potential: 0 V
- Duration: 5 s
- Repeat 10 times
    - Potential step 1: 1.2 V, duration: 2.5 s
    - Potential step 2: 0 V, duration: 2.5 s

Figure 3.25 shows an overview of the Chrono charge discharge procedure.

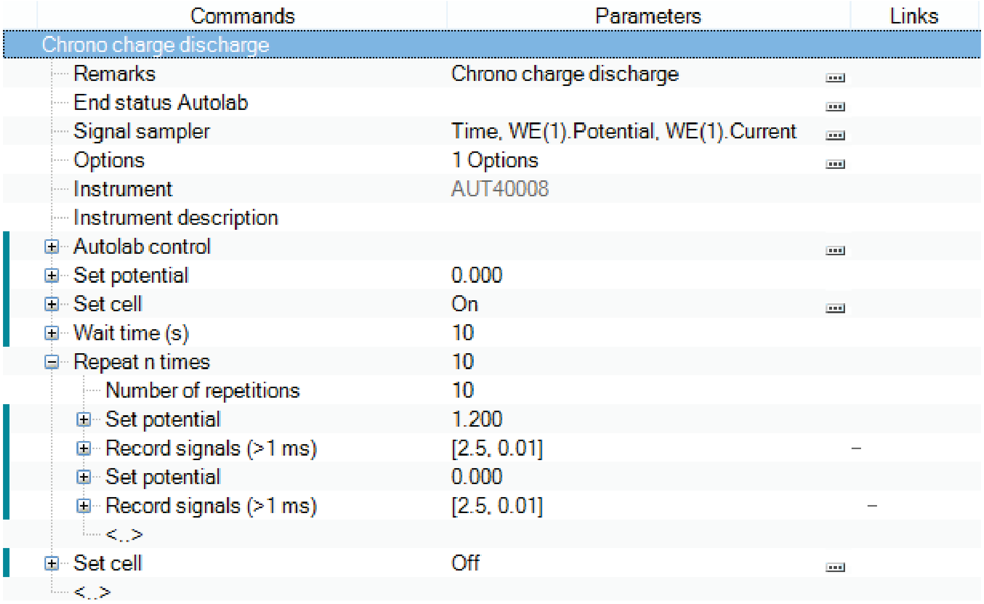


Figure 3.25 – The Autolab Chrono charge discharge procedure

The signals sampled during this procedure are:

- Corrected time
- Time
- WE(1).Potential
- WE(1).Current
- Index

Figure 3.26 shows a measurement on the dummy cell (a) with the Autolab Chrono charge discharge procedure.

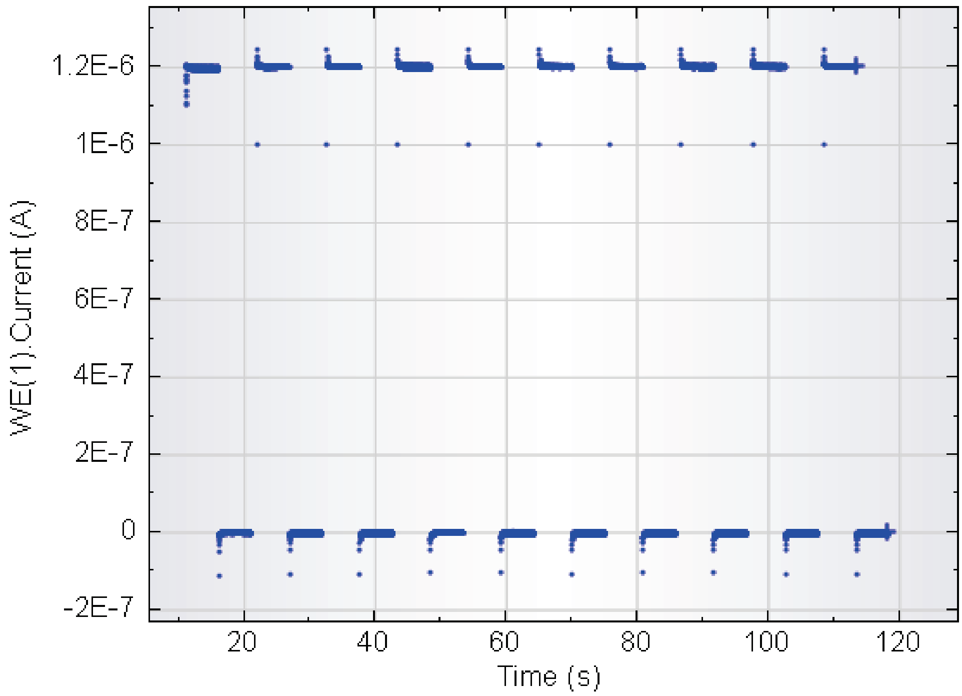


Figure 3.26 – The measured data obtained with the standard dummy cell (a) with the Chrono charge discharge procedure

3.21 – i-Interrupt

Hardware Tags	None
Profile Tags	Intermediate
Application Tags	None

This procedure can be used to perform a current interrupt measurement in order to determine the value of the uncompensated resistance.

Note

This procedure cannot be used in combination with the PGSTAT10 and the μAutolab type II/III.

More information about the use of this procedure is provided in the **iR compensation tutorial**, available from the Help menu in NOVA.

3.22 – i-Interrupt high speed

Hardware Tags	ADC10M or ADC750 module
Profile Tags	Intermediate
Application Tags	None

This procedure is similar to the i-Interrupt procedure. This procedure uses the optional fast sampling ADC module (ADC750 or ADC10M).

This procedure can be used to perform a current interrupt measurement in order to determine the value of the uncompensated resistance.

Note

This procedure cannot be used in combination with the PGSTAT10 and the μAutolab type II/III. This procedure requires the fast sampling ADC module.

More information about the use of this procedure is provided in the **iR compensation tutorial**, available from the Help menu in NOVA.

3.23 – Positive feedback

Hardware Tags	None
Profile Tags	Intermediate
Application Tags	None

The Positive feedback procedure provides the means to determine the value of the uncompensated resistance using the positive feedback method.

Note

This procedure cannot be used in combination with the PGSTAT10 and the μAutolab type II/III.

More information about the use of this procedure is provided in the **iR compensation tutorial**, available from the Help menu in NOVA.

3.24 – FRA impedance potentiostatic

Hardware Tags	FRA32M or FRA2 module
Profile Tags	Basic
Application Tags	Corrosion, Energy, Interfacial electrochemistry, Semiconductors

The FRA impedance potentiostatic procedure requires the optional FRA32M or FRA2 impedance analyzer module. This procedure can be used to perform a potentiostatic frequency scan to determine the electrochemical impedance of the cell.

More information about the use of the FRA32M or FRA2 module is provided in the **Impedance tutorial**, available from the Help menu in NOVA.

3.25 – FRA impedance galvanostatic

Hardware Tags	FRA32M or FRA2 module
Profile Tags	Basic
Application Tags	Energy, Semiconductors

The FRA impedance galvanostatic procedure requires the optional FRA32M or FRA2 impedance analyzer module. This procedure can be used to perform a galvanostatic frequency scan to determine the electrochemical impedance of the cell.

More information about the use of the FRA32M or FRA2 module is provided in the **Impedance tutorial**, available from the Help menu in NOVA.

3.26 – FRA potential scan

Hardware Tags	FRA32M or FRA2 module
Profile Tags	Intermediate
Application Tags	Semiconductors

The FRA potential scan procedure requires the optional FRA32M or FRA2 impedance analyzer module. This procedure can be used to perform a potentiostatic frequency scan at different DC potentials to determine the electrochemical impedance of the cell for each DC potential value.

More information about the use of the FRA32M or FRA2 module is provided in the **Impedance tutorial**, available from the Help menu in NOVA.

The Autolab Potentiostat/Galvanostat

Nova can be used to control Autolab potentiostats/galvanostats with USB interface[35]. While the technical specifications of each of the instruments might be different, the operating principle remains the same.

This chapter provides an overview of the Autolab as well as information concerning the digital nature of the instrument. Information regarding noise issues is also provided.

4 – Autolab Hardware information

4.1 – Overview of the Autolab instrument

The Autolab instrument combined with the software is a computer-controlled electrochemical measurement system. It consists of a data-acquisition system and a potentiostat/galvanostat (see Figure 4.1).

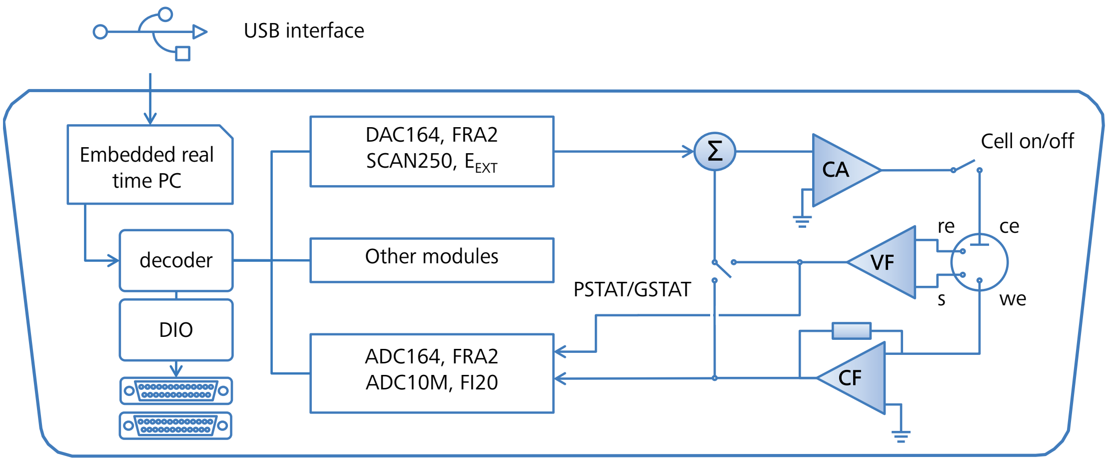


Figure 4.1 – Overview of the Autolab potentiostat/galvanostat

The Autolab has the following key digital components:

- USB interface
- Embedded real-time PC
- Decoder and DIO controller

[35] Except the PSTAT10 and the µAutolab type I.

The digital components are interfaced through the Autolab modules to the analog potentiostat/galvanostat circuit. The latter consists of the following components:

- The summation point (Σ)
- The control amplifier (CA)
- The voltage follower (VF)
- The current follower (CF)

The summation point (Σ) is an adder circuit that feeds the input of the control amplifier. It is connected to the output of the several key modules of the Autolab:

- DAC164
- FRA[36] DSG
- SCAN250[37]
- E_{in}

Figure 4.2 shows a schematic overview of the different connections to the summation point of the Autolab potentiostat/galvanostat[38]. The labels shown in Figure 4.2 correspond to the dividing factors used for each signal on the summation point. For example, the signal generated by the FRA module (FRA-DSG) has a maximum amplitude of 3.5 V (RMS), which is divided by 10 at the input of the summation point, resulting in an effective maximum amplitude of 0.35 V (RMS).

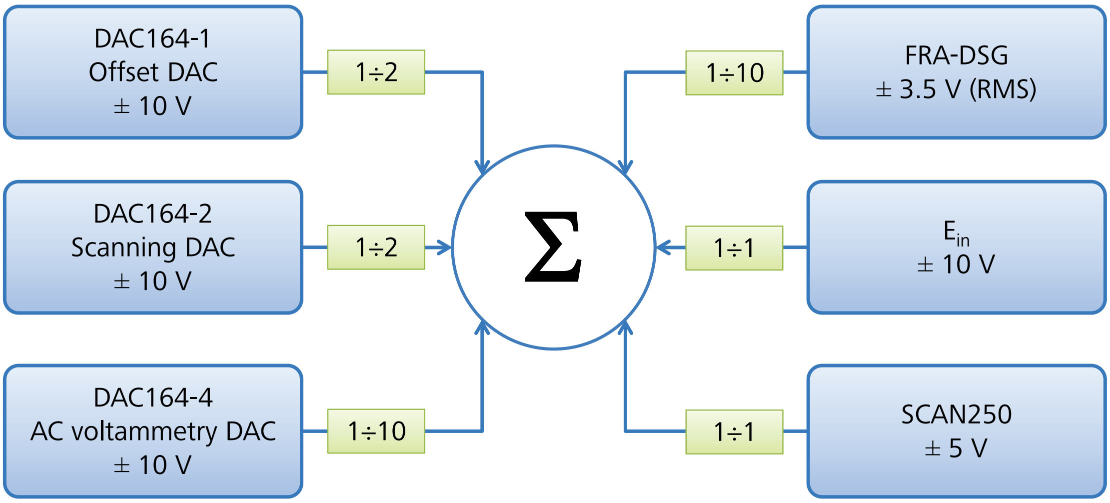


Figure 4.2 – Mapping of the inputs of the summation point

[36] This input is connected to the FRA32M or the FRA2 module.
[37] Or earlier version SCANGEN.
[38] Offset DAC, SCAN250/SCANGEN and E_{in} are not available on the μAutolab III. SCAN250/SCANGEN, FRA-DSG and E_{in} are not available on the PGSTAT101. SCAN250/SCANGEN and E_{in} are not available on the M101 module.

The control amplifier provides the output voltage on the counter electrode (CE) with respect to the working electrode (WE) required to keep the potential difference between the reference electrode (RE) and the sense (S) at the user defined value, in potentiostatic mode, or the user required current between the counter electrode (CE) and the working electrode (WE) in galvanostatic mode.

The output of the control amplifier can be manually or remotely disconnected from the electrochemical cell through a cell ON/OFF switch. The voltage follower (VF) is used to measure the potential difference between the reference electrode and the sense and the current follower (CF). The current follower has several current ranges providing different current-to-voltage conversion factors.

The output of the CF and the VF are fed back to the analog-to-digital converter modules of the Autolab:

- ADC164
- FRA ADC[39]
- ADC10M[40]
- FI20

Furthermore, the output of the VF or the CF is fed back to the summation point to close the feedback loop in potentiostatic or galvanostatic mode, respectively.

The ADC164 provides the possibility of measuring analog signals. The input sensitivity is software-controlled, with ranges of ± 10 V (gain 1), ± 1 V (gain 10) and ± 0.1 V (gain 100). The resolution of the measurement is 1 in 65536 (16 bits, ADC164). Analog signals can be measured with a rate of up to 60 kHz. The ADC164 is used to measure the output of the Voltage Follower (VF) and Current Follower (CF) of the potentiostat/galvanostat module.

The DAC164 generates analog output signals. The output is software-controlled within a range of ± 10 V. The resolution of the DAC164 is 1 in 65535 (300 µV). In the Autolab PGSTAT two channels of the DAC are used to control the analog input signal of the potentiostat/galvanostat. The µAutolab only uses one DAC channel to control the analog input (see Figure 4.2). The values of the DACs are added up in the potentiostat and divided by 2. DAC channel 1 is used as a variable DAC and DAC channel 2 provides a fixed offset. This results in an output of ± 10 V with a resolution of 150 µV.

In practice this means that the potential range available with the Autolab PGSTAT during an electrochemical experiment is ± 5 V with respect to the offset potential generated by the offset DAC (DAC164-1). The available potential range is therefore -10 V to 10 V with the Autolab PGSTAT and -5 V to 5 V with the µAutolab.

[39] These inputs are connected to the FRA32M or the FRA2 module.
[40] Or earlier version ADC750.

One additional DAC164 channel is available on the PGSTAT N series, DAC164-4. This channel is hardwired to the summation point and it is divided by 10. This input is used for measurements involving a small amplitude modulation (like AC voltammetry and AC voltammetry second harmonic). Presently, these methods and the use of this channel is not yet implemented in NOVA. The connection to the summation point can be removed if necessary[41].

The DIO-part offers the possibility of controlling electrode systems, motorburettes or other equipment that can be controlled by TTL signals. This module can also be used to send or receive trigger signals to or from TTL devices. If an automatic mercury electrode such as PAR303 or Metrohm 663 VA Stand is used, gas purging and drop time can be activated. The interface for mercury electrodes, called IME303 or IME663, provides all necessary signals and connections for these electrodes, as well as for a drop knocker of a dropping mercury electrode (only for IME303).

The embedded PC can be in two different locations, depending on the type of interface:

- Inside of the Autolab-USB Interface box
- Inside of the Autolab-USB instrument

[41] Contact your Autolab distributor for more information.

4.1.1 – Event timing in the Autolab

The embedded PC is equipped with a 1 MHz timer that is used by the software to control the timing of events during measurements. The shortest interval time on the embedded PC is 1 µs. When a procedure is started in NOVA, the procedure is first uploaded from the host PC to the embedded PC, through the USB connection. The measurement can then be started.

Depending on the type of command that NOVA encounters during the measurements two timing protocols are used:

1. **Measurement commands:** all measurement commands in NOVA are *Timed* commands. Whenever NOVA encounters a measurement command, it will be executed using the timing provided by the embedded computer of the Autolab. If several measurement commands are located in sequence, the sequence is executed without interruption. This ensures that the measurement commands in the sequence are executed with the smallest possible time gap. The actual time difference between two consecutive commands depends on the hardware changes required during the transition between the two commands. Switching current ranges or using the cell switch are time consuming steps since they involve mechanical relays which require a fixed settling time. Taking into account these hardware defined interval times, the effective time gap between two consecutive commands in a Timed procedure will be ≤ 10 ms.

Note

Measurement commands are identified by a green line on the left-hand side of the procedure editor.

2. **Host commands:** all the other commands in NOVA are host commands. These commands are executed by the host PC using the timing provided by this computer. Since the host PC is also involved in other Windows activity, accurate timing of events cannot be guaranteed and the effective interval time between two consecutive host commands will depend entirely on the amount of activity on the host PC. Depending on the command sequence, the time gap can be as short as ~ 2 s (transition between host command to measurement command) or several seconds (transition between measurement command and host command). Transfer of large amounts measured data points is particularly time consuming[42].

[42] The on-board memory of the fast sampling ADC module (ADC10M or ADC750) can store up to one million data points. Allow for gap times of several seconds when large data sets are transferred from the Autolab to the host computer.

Figure 4.3 shows the Autolab *Linear polarization* procedure, in which measurement and host commands are identified using the green timing guide on left-hand side.

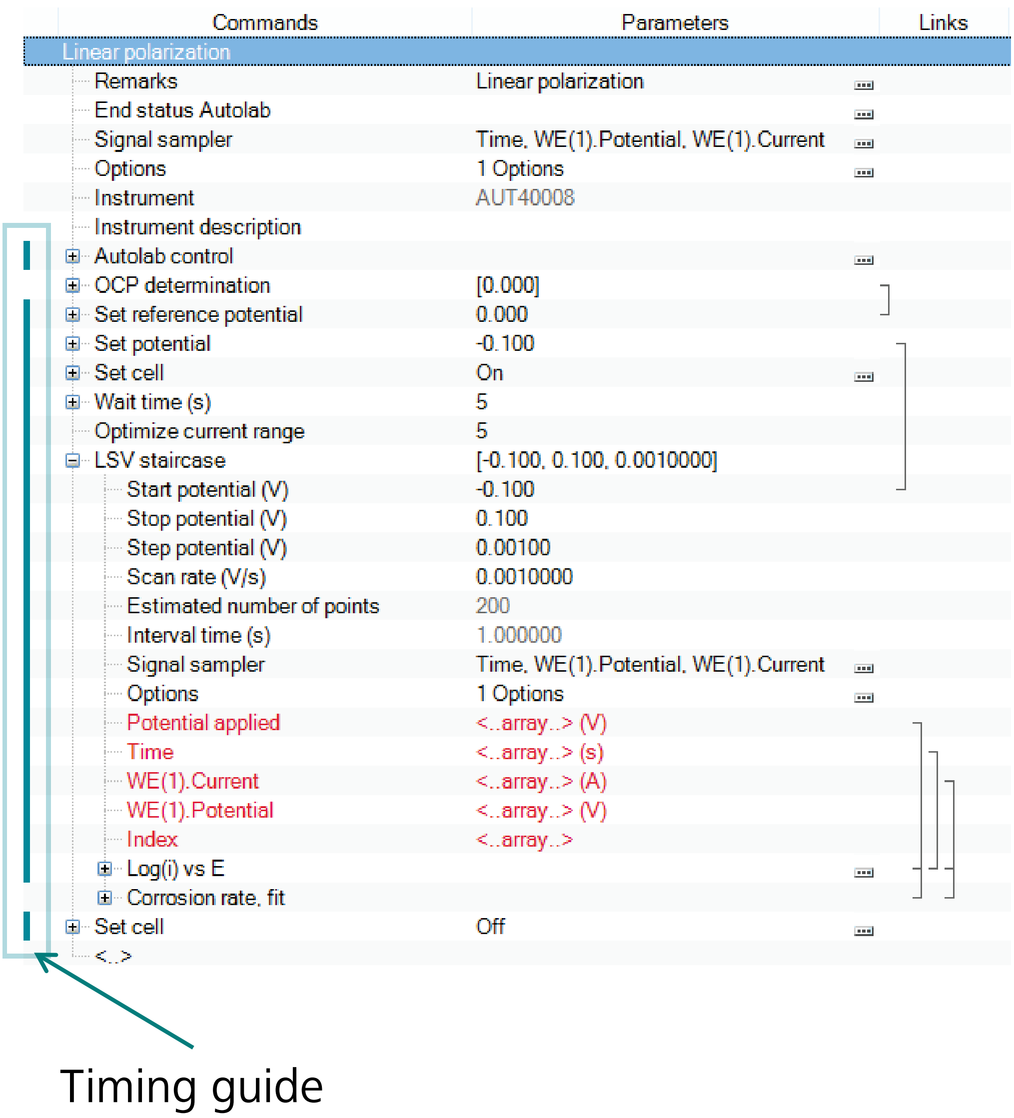


Figure 4.3 – The Autolab *Linear polarization* procedure (with the timing guide highlighted)

The procedure contains a series of measurement commands which are interrupted at two commands:

- OCP determination command
- Corrosion rate, fit

These interruptions are indicated in the timing guide shown in Figure 4.3 by the matching breaks located right next to the two commands.

The *OCP determination* command is a host command because it requires the derivative of the measured OCP to be calculated during the measurement. The *Corrosion rate, fit* command is, like all analysis commands, a host command.

The timing guide indicates that when this procedure is performed, a small interruption can be expected when the OCP determination command and when the Corrosion rate, fit command are executed.

Note

If needed, the *Corrosion rate, fit* command can be moved to the end of the procedure to prevent the interruption between the *LSV staircase* command and the *Set cell* command.

4.2 – Consequence of the digital base of the Autolab

It is clear that the digital nature of the instrument has consequences for the measurements. The consequences for the different techniques are[43]:

- The minimum potential step or pulse in all techniques is 150 µV (16 Bit DAC164).
- All potential steps are rounded up or down to the nearest possible multiple of 150 µV.
- In cyclic voltammetry staircase, the interval time, Δt, or time between two consecutive steps is given by:

$$\Delta t = \frac{E_{step}}{\vec{v}}$$

Where E_{step} is the potential step and $\vec{v}$ is the scan rate in V/s.

The response of the electrochemical cell is recorded digitally. Therefore the resolution of the measurements is also limited. The actual resolution depends on the technique and on the amplitude of the signal. Since the A/D converter is equipped with a software programmable amplifier, the absolute resolution depends on the gain of the amplifier. The gains used are 1, 10 and 100 times the input signal.

NOVA automatically selects the best possible gain during a measurement. Gain 10 and 100 are used when the signal is small enough.

When the absolute value of the current is higher than (0.5 * current range), the resolution of the current measurement equals:

$$\frac{C.R.\cdot 20}{2^{16}\cdot 1} = C.R.\cdot 0.0003$$

When the absolute value of the current is lower than (0.5 * current range), the resolution equals:

[43] The same applies for Galvanostatic control of the instrument.

$$\frac{C.R.\cdot 20}{2^{16}\cdot 10} = C.R.\cdot 0.00003$$

When the absolute value of the current is lower than (0.05 * current range), the resolution equals:

$$\frac{C.R.\cdot 20}{2^{16}\cdot 100} = C.R.\cdot 0.000003$$

The effect of the limited resolution can be seen, for instance when low currents are measured at a high current range. In such cases a lower current range has to be applied, if possible. When automatic current ranging is used, the most suitable current range is selected automatically.

Care must be taken when using this option in the following situations:

- High frequency square wave voltammetry is applied.
- High scan rates in cyclic and linear sweep voltammetry are applied.

Switching of the current range takes about 0.5 ms to 2 ms. Therefore an erroneous point can be measured when the current range is switched. Most of the time, this error can be corrected by smoothing the plot afterwards.

4.3 – Autolab PGSTAT information

This section provides specific information for the Autolab PGSTAT series of instruments. The following instruments fall under this category: PGSTAT12, 128N, 30, 302, 302N, 100 and 100N[44].

4.3.1 – Front panel and cell cable connection

There are four connectors on the front panel of the PGSTAT. The cable that connects to the WE and CE should be plugged into the WE/CE socket while the cable with the differential amplifier (leading to the RE, S and optionally WE2 electrodes) connects to the RE/S socket. A ground cable, embedded in the WE/CE cable connection, can be used to plug to the earth bulkhead for shielding purposes. Finally a monitor cable can be connected to a dedicated connector (see Figure 4.4).

[44] For information on the PGSTAT302F, please refer to Section 4.4. For information on the PGSTAT101 and the Multi Autolab with M101 module, please refer to Section 4.5. For information on the µAutolab type II and III, please refer to Section 4.6.

Note

The Series 7 instruments and early Series 8 instruments are provided with an additional ground cable which should be connected to the plug provided above the connector for the monitor cable. This ground connector should be used for grounding purposes.

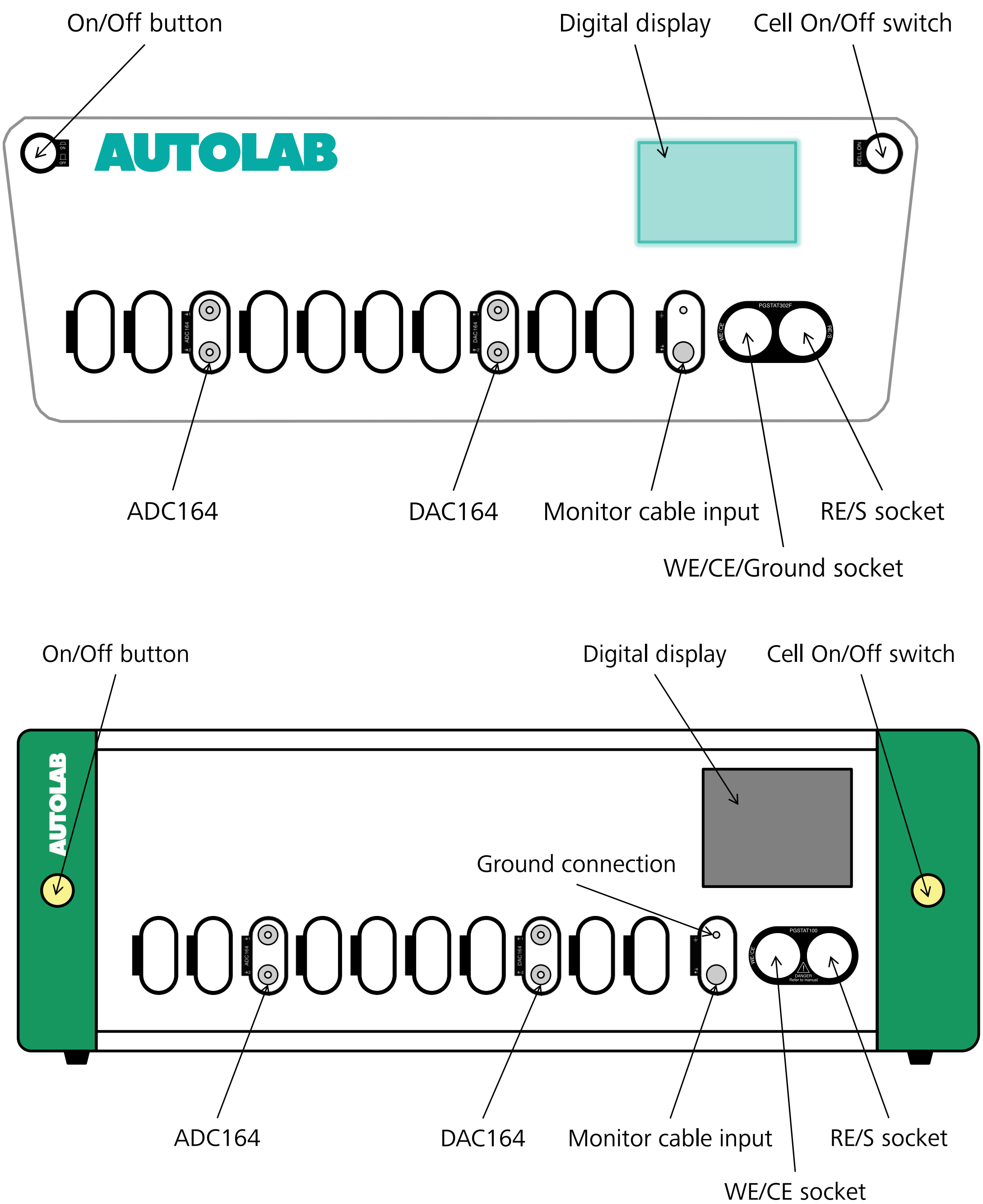


Figure 4.4 – Overview of the Autolab PGSTAT (top –Series 8 PGSTAT, bottom – Series 7 PGSTAT)

Note

The Series 8 instruments are provided with an additional ground cable embedded into the CE/WE cable. This ground connector should be used for grounding purposes.

The cell cables are labelled as follows:

- Working or indicator electrode, WE (red)
- Sense electrode, S (red)
- Reference electrode, RE (blue)
- Auxiliary or counter electrode, CE (black)

In a four electrode setup, each of the cell cable connectors is used independently. In a three electrode set-up the working electrode and sense lead are both connected to the working electrode. In a two electrode set-up the counter and reference electrode lead are both connected to the same electrode (see Figure 4.5).

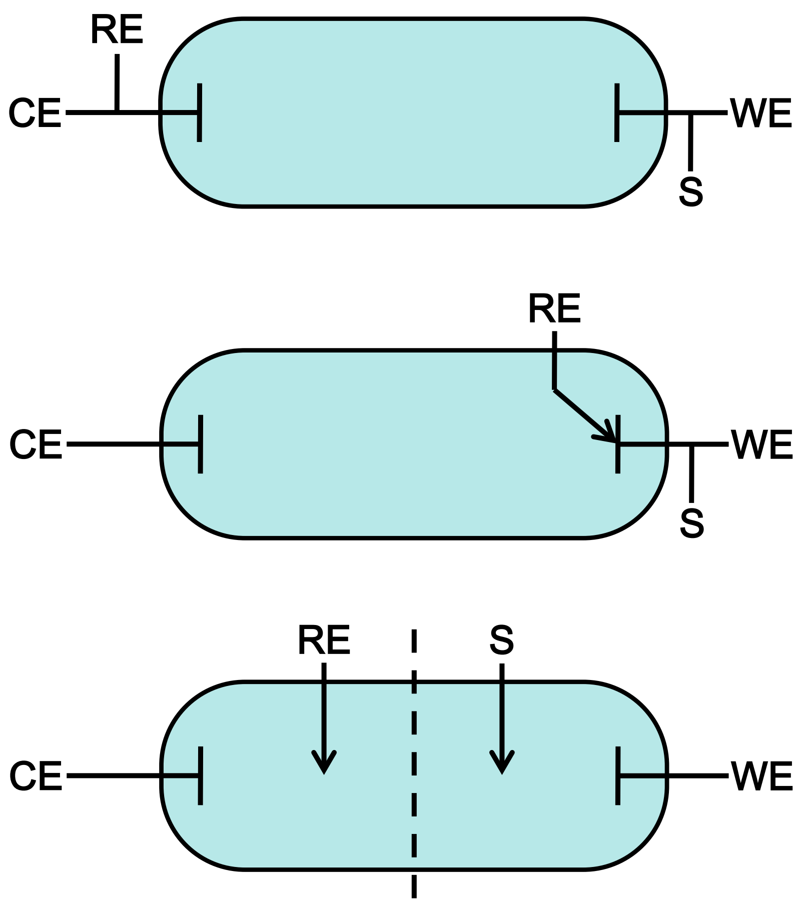


Figure 4.5 – Overview of the possible cell connections with the Autolab PGSTAT (from top to bottom: two electrode, three electrode and four electrode setup)

4.3.2 – Power up

The settings of the PGSTAT on power-up are pre-defined. The following settings are used:

- Cell: off
- Mode: Potentiostatic
- Bandwidth: High stability
- iR Compensation: off
- Current range: 10 mA
- ECD mode: off, if applicable

4.3.3 – Connections for analog signals

The Autolab PGSTAT provides connections for analog signals through two different types of connectors:

- BNC connectors directly located on the front panel of the instrument
- BNC connectors located on the monitor cable

4.3.3.1 – Connections for analog signals (front panel)

The ADC164 module and the DAC164 module are fitted with two analog inputs and two analog outputs, respectively (see Figure 4.6).

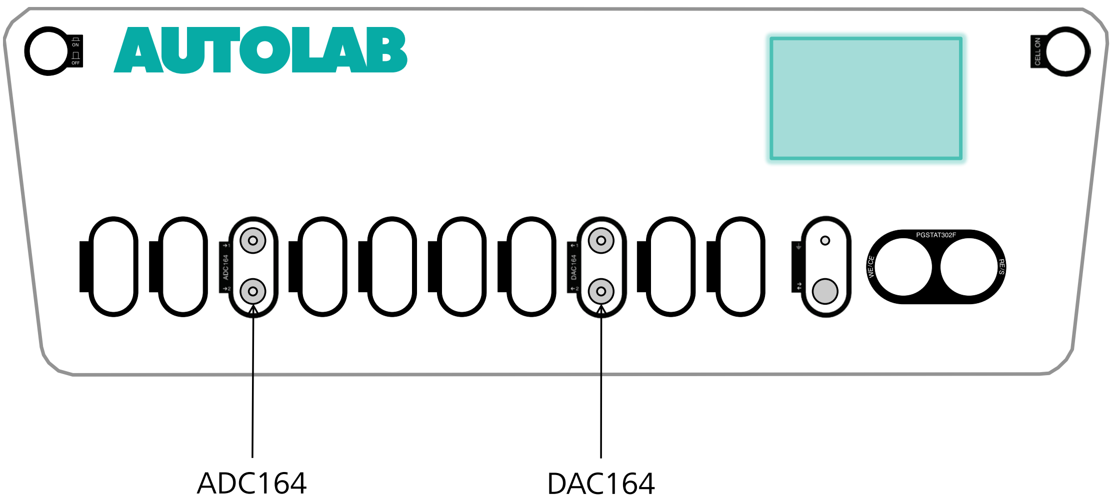


Figure 4.6 – Overview of the connections for analog signals provided on the front panel of the Autolab PGSTAT (ADC164 and DAC164)

ADC164 – The ADC164 inputs, labelled →1 and →2 on the front panel, can be used to record any analog signal with a ± 10 V value range. The input impedance of the two analog inputs is 50 Ω.

DAC164 – The DAC164 outputs, labelled ←1 and ←2 on the front panel, can be used to generate any analog signal with a ± 10 V value range. The output impedance of these two inputs is 50 Ω. Corrections should be made with loads < 100 kΩ. Because of dissipation, the minimum load impedance should be 200 Ω.

Note

The DAC164 ←1 and ←2 outputs are identified as DAC channels 3 and 4, respectively in the *Set DAC* command.

4.3.3.2 – Connections for analog signals (monitor cable)

With the supplied monitor cable, there are a number of BNC connectors to the PGSTAT analog circuits (see Figure 4.7). All the signals are with respect to Autolab ground and indirectly to protective earth. Avoid creating ground loops as this will often degrade the performance of the PGSTAT.

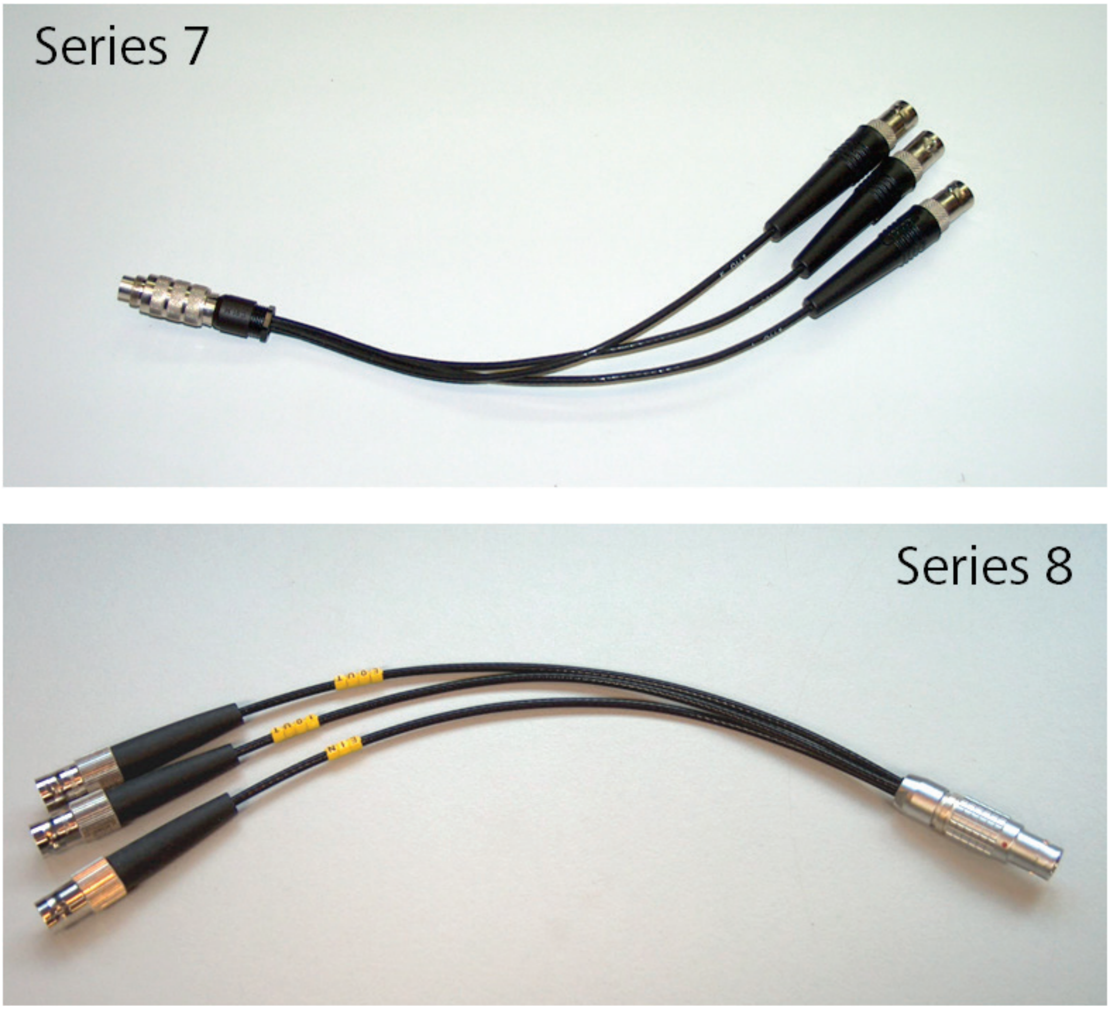


Figure 4.7 – The monitor cable for the Series 8 and the Series 7 PGSTAT

The following signals are available:

E_{OUT} – This output corresponds to the differential potential of RE versus S[45]. The output voltage will vary between ±10 V. The output impedance is 50 Ω, so a correction should be made if a load < 100 kΩ is connected. The minimum load impedance is 200 Ω.

i_{OUT} – This signal corresponds to the output of the current-to-voltage converter circuit of the PGSTAT. A 1 V signal corresponds to {1 x the selected current range}. The output level varies between ± 10 V. The output impedance is 50 Ω, so a correction should be made if a load < 100 kΩ is connected. The minimum load impedance is 200 Ω.

E_{IN} – This is an analog voltage input, that can only be used after it has been enabled in software, using the *Autolab control* command (see Figure 4.8). Do not leave it enabled unnecessarily, to prevent noise pickup by the system. This input is directly connected to the summation point, Σ, of the PGSTAT. In PSTAT mode, a

[45] The E_{out} value corresponds to -WE(1).Potential.

1 V signal will add 1 V to the cell voltage, while in GSTAT mode a 1 V signal adds an extra current of {1 x the selected current range} to flow. In both cases, the external signal adds to any pre-defined voltage or current. The input voltage range is ± 10 V. Input impedance is 1 kΩ (only when input is activated) so a correction should be made when the source impedance is > 1 Ω.

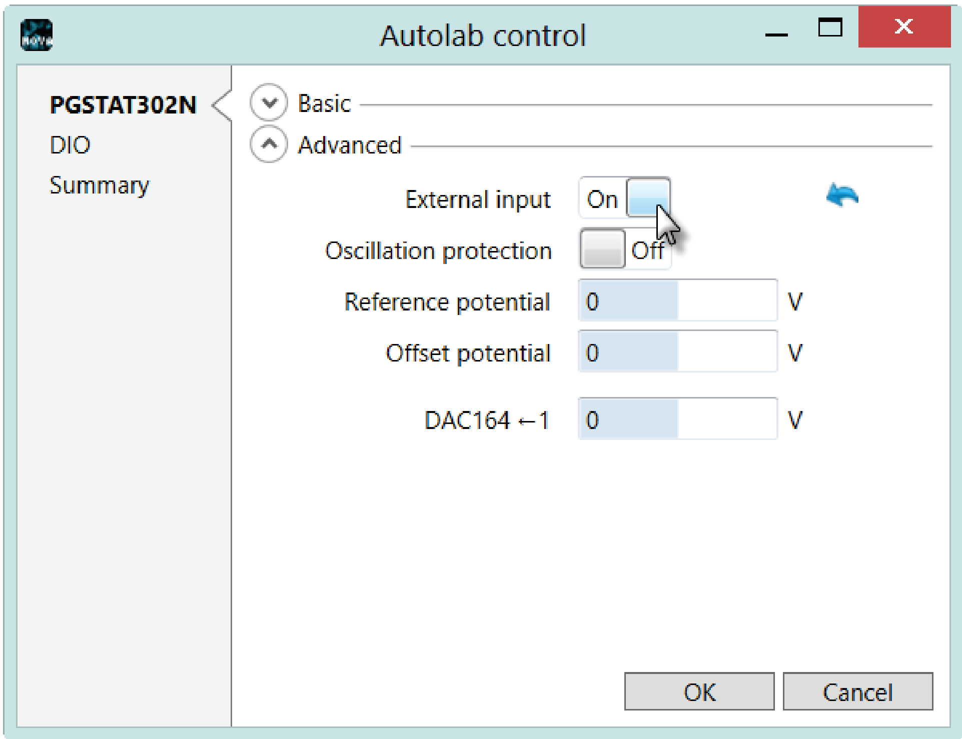


Figure 4.8 – The external input is enabled in the Autolab control window

4.3.4 – High stability, High speed and Ultra high speed

The PGSTAT is equipped with three different bandwidth settings: High stability (HSTAB), High speed and Ultra high speed. The bandwidth can be defined using the Autolab control command (see Figure 4.9).

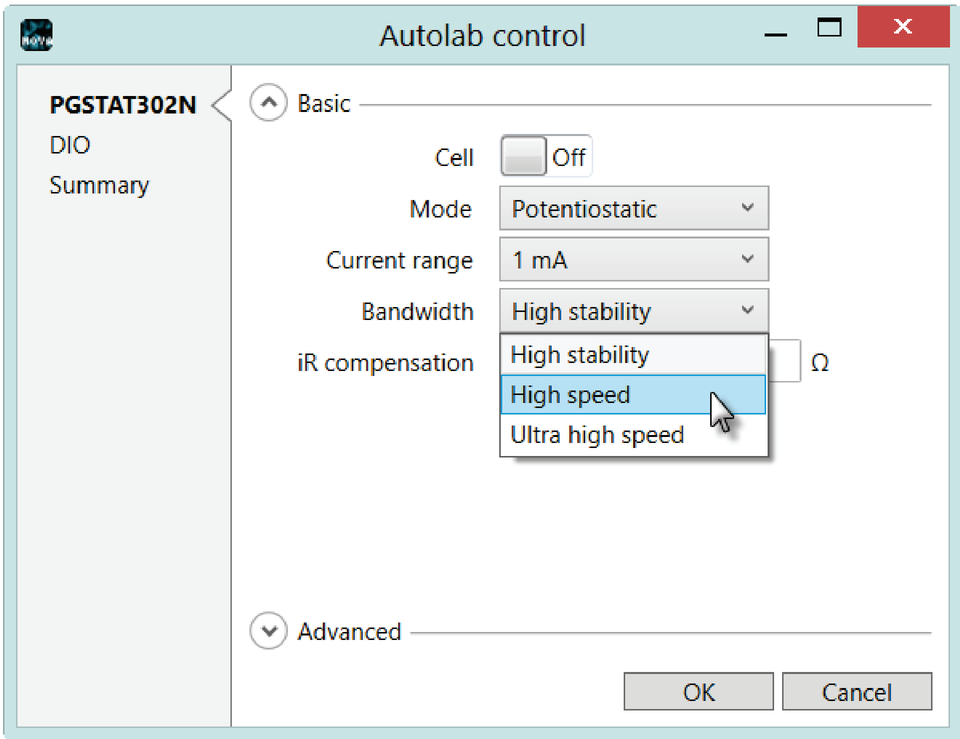


Figure 4.9 – The Autolab control window can be used to set the bandwidth of the PGSTAT

The purpose of these different modes of operation is to provide a maximum bandwidth, maintaining stability in the PSTAT or GSTAT control loop. The normal mode of operation is High stability[46]. This gives the Control Amplifier a bandwidth of 12.5 kHz. The HSTAB indicator on the front panel of the PGSTAT and in the Autolab display is lit when the High stability mode is active (see Figure 4.10).

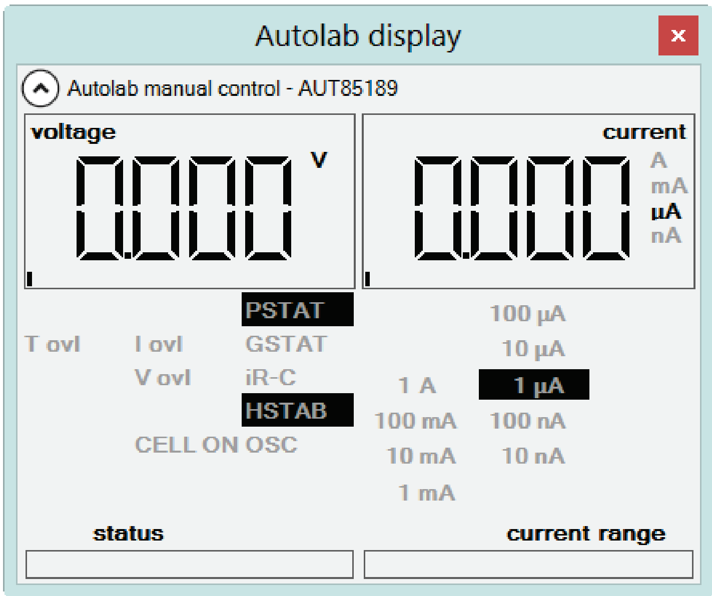


Figure 4.10 – A HSTAB indicator is provided on the Autolab display

[46] Power up default setting.

This setting is the most appropriate for measurements at low frequencies or low scan rates. The noise in the i and E signals will be minimized. Measurements at high frequency or at high scan rates require a faster mode of operation.

When operating in High speed mode, the control amplifier will have its bandwidth extended with one decade up to 125 kHz. Some cells can show ringing or oscillation using this setting, particularly highly capacitive cells in PSTAT mode. Increasing the bandwidth also increases the noise levels for the i and E signals. The High speed mode is automatically selected during impedance measurement at frequencies > 10 kHz.

Note

It is possible to switch from High stability to High speed by clicking the HSTAB label in the Autolab display. In High speed mode, this label will be unlit, both on the front panel of the PGSTAT and on the Autolab display. Clicking the HSTAB label again switches the bandwidth back to High stability.

For applications requiring very high bandwidth, the Ultra high speed mode can be selected. In this mode, the control amplifier bandwidth is extended to 500 kHz (PGSTAT12, PGSTAT128N, PGSTAT100 and PGSTAT100N) or 1.25 MHz (PGSTAT30, PGSTAT302 and PGSTAT302N). There is a significant oscillation risk using this setting, and the noise levels will generally show an increase relative to the High speed or High stability mode. The Ultra high speed mode is automatically selected during impedance measurements at frequencies > 100 kHz, while the High stability mode is selected for frequencies below 10 kHz (see Figure 4.11).

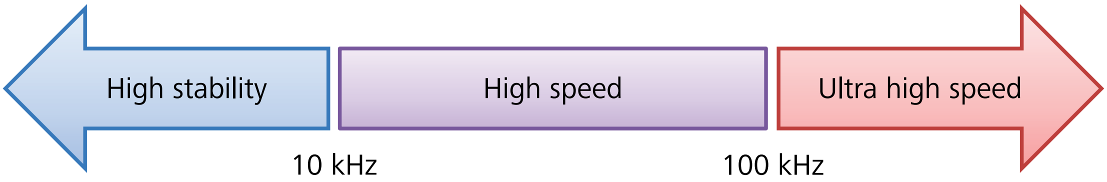


Figure 4.11 – Bandwidth limits in the Autolab PGSTAT

Warning

The higher the bandwidth, the more important it is to pay attention to adequate shielding of the cell and the electrode connectors. The use of a Faraday cage is recommended in this case.

4.3.5 – RE input impedance and stability

The electrometer RE input contains a small capacitive load. If the capacitive part of the impedance between CE and RE is comparatively large, phase shifts will occur which can lead to instability problems when working in potentiostatic mode. If the

impedance between the CE and the RE cannot be changed and oscillations are observed, it is recommended to select the High stability mode to increase the system stability. In general, the use of High stability leads to a more stable control loop, compared to High speed or Ultra high speed and a significantly lower bandwidth.

To make use of the full potentiostat bandwidth (Ultra high speed mode), the impedance between CE and RE has to be lower than 35 kΩ[47]. This value is derived by testing. In galvanostat mode, this large impedance between CE and RE, will usually not lead to stability problems, because of the current feedback regulation.

4.3.6 – Galvanostatic FRA measurements

The capacitive part of the impedance between RE and ground is an important aspect to consider when performing FRA measurements in galvanostat mode. Large reference electrode impedance values may introduce a phase shift at low frequencies. The origin of the phase shift between the CE and the RE cannot be determined from the FRA data.

Galvanostatic FRA measurements at 1 MHz require a maximum of 3 kΩ reference electrode impedance to keep phase errors within the ± 5 ° limit.

4.3.7 – Galvanostat, potentiostat and iR-compensation bandwidth

For galvanostatic measurements on low current ranges, the bandwidth limiting factor becomes the current-to-voltage circuit rather than the control amplifier.

For stability reasons it is not recommended to use the High speed mode for current ranges < 10 µA. The Ultra high speed mode is also not recommended for current ranges < 1 mA.

As the current measurement circuit plays an important role in the iR compensation technique, its use is also subject to bandwidth limitations.

[47] Empirical value.

A general indication of the maximum available bandwidth for GSTAT and for iR compensation can be found in Table 4.1:

Instrument	PGSTAT12/128N/100/100N		PGSTAT30/302/302N	
Mode	GSTAT	iR/C – PSTAT	GSTAT	iR/C – PSTAT
1 A – 1 mA	> 500 kHz	> 500 kHz	> 1.25 MHz	> 1.25 MHz
100 µA	125 kHz	500 kHz	125 kHz	1 MHz
10 µA	100 kHz	100 kHz	100 kHz	100 kHz
1 µA	10 kHz	10 kHz	10 kHz	10 kHz
100 nA	1 kHz	1 kHz	1 kHz	1 kHz
10 nA	100 Hz	100 Hz	100 Hz	100 Hz

Table 4.1 – Bandwidth overview for the different instruments

At the same time, the iR-compensation bandwidth limits indicate up to which frequency current measurements can be made in potentiostatic mode (either with or without iR compensation).

4.3.8 – Galvanostatic operation and current range linearity

For galvanostatic experiments, automatic current ranging is not possible. The measurements are performed in a fixed current range. Each current range on the instrument is characterized by a specific linearity limit and this specification determines the maximum current that can be applied in galvanostatic mode.

The linearity limitation also applies on measurements performed in potentiostatic mode in a fixed current range.

Table 4.2 provides an overview of the current range linearity for the different PGSTAT instruments.

Current range	PGSTAT12 PGSTAT100 PGSTAT100N	PGSTAT128N	PGSTAT30	PGSTAT302 PGSTAT302N
1 A	n.a.	0.8	1	2
100 mA	2.5	3	3	3
10 – 1 mA	3	3	3	3
100 – 1 µA	3	3	3	3
100 – 10 nA	3	3	3	3

Table 4.2 – Linearity limit for the different instruments

For example, in the 100 mA current range, the maximum current that can be applied, galvanostatically, using the PGSTAT302N, is 300 mA. The maximum current that can be measured in the 100 mA current range, using the same instrument is 1000 mA, although currents exceeding 300 mA will be measured outside of the linearity limit of this current range.

In galvanostatic operation, the applied current values are checked during the procedure validation step. When the applied current exceeds the linearity limit for the specified current range, an error message will be shown in the procedure validation screen (see Figure 4.12).

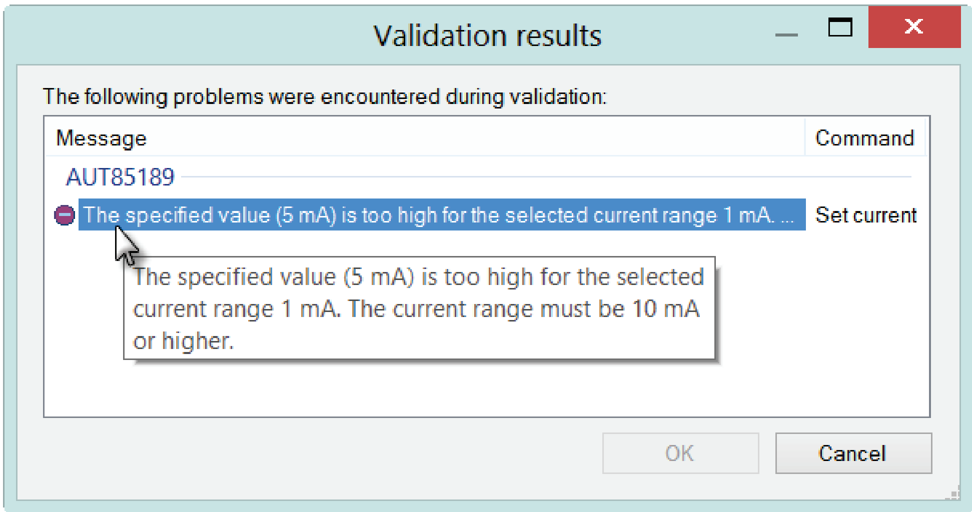


Figure 4.12 – The procedure validation step always checks the applied current values for the allowed linearity

> **Note**
>
> In potentiostatic mode, this check is not performed. It is possible to measure a current value in a fixed current range, even if the current value exceeds the linearity limit of the active current range. This triggers a current overload warning. When this happens during a measurement, a message will be shown in the user log, suggesting a modification of the current range (see Figure 4.13).

User log message	Time	Date	Command
Autolab/USB connected (AUT85189)	4:05:04 PM	1/15/2013	-
Overload occurred in 1 µA current range, use a higher current range.	4:10:43 PM	1/15/2013	CV staircase

Figure 4.13 – When a current overload is detected, a suggestion is shown in the user log

4.3.9 – Oscillation detection

The PGSTAT has a detector for large-amplitude oscillation. The detector will spot any signal swing that causes the control amplifier to produce both a positive and a negative Voltage overload within ~ 200 µs. Thus, large oscillations at frequencies > 2.5 kHz will be detected. Upon oscillation, the OSC indicator on the PGSTAT front panel will be activated. The V_{ovl} warning will also be shown in the Autolab display. An oscillation protection feature can be enabled or disabled in the software, using the *Autolab control* command (see Figure 4.14).

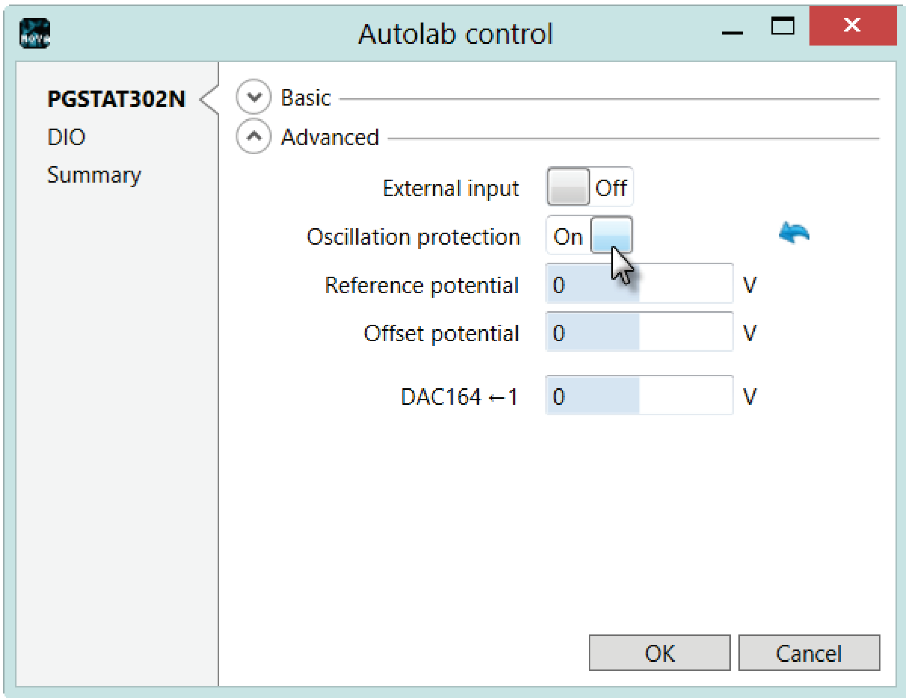


Figure 4.14 – The Autolab control window can be used to switch the oscillation protection on or off

If the oscillation protection is enabled, the occurrence of oscillation will also immediately turn off the manual cell switch of the Autolab. When this happens, both the OSC indicator and the manual cell switch start blinking. The Autolab display will show the message ‘Cell manually off’ (see Figure 4.15).

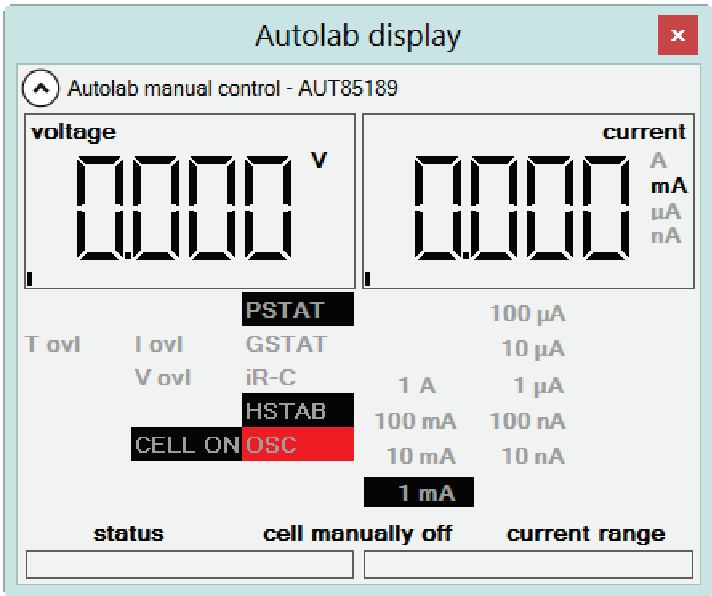


Figure 4.15 – The cell manually off is displayed when the oscillation protection circuit is triggered

The cell may be switched on again by pressing the manual cell switch button. If oscillation resumes, the cell switch will be turned off as soon as the button is released. Holding the button pressed in, provides an opportunity to observe the system during oscillation.

Some cells that cause ringing when switching the cell on or changing the current range can falsely trigger the oscillation detector. If this happens, the Oscillation protection may be switched off in the software in order to prevent an accidental disconnection of the cell.

4.3.10 – Maximum reference electrode voltage

The differential electrometer input contains an input protection circuitry that becomes active after crossing the ±10 V limit. This is implemented to avoid electrometer damage. Please note that the V_{ovl} indicator will not light up for this type of voltage overload. The measured voltage will be cutoff at an absolute value of 10.00 V.

Depending on the cell properties, galvanostatic control of the cell could lead to a potential difference between the RE and the S/WE larger than 10 V. This situation will trigger the cutoff of the measured voltage to prevent overloading the differential amplifier.

4.3.11 – Active cells

Some electrochemical cells such as batteries and fuel cells are capable of delivering power to the PGSTAT. This is allowed only to a maximum ‘cell’ power, P_{MAX}. This value depends on the instrument (see Table 4.3).

Instrument	Maximum power, P_{MAX} (W)
PGSTAT12	2.5
PGSTAT128N	8
PGSTAT30	10
PGSTAT302/302N	20
PGSTAT100/100N	2.5

Table 4.3 – Maximum power rating for the different PGSTAT models

This means that cells showing an absolute voltage ($|V_{cell}|$) of less than 10 V between WE and CE are intrinsically safe. They may drive the PGSTAT output stage into current limit but will not overload the amplifier. On the other hand, cells that have an absolute voltage higher than 10 V between WE and CE may only deliver a maximum current, i_{MAX} given by:

$$i_{MAX} = \frac{P_{MAX}}{|V_{MAX}|}$$

4.3.12 – Grounded cells

The measurement circuitry of the Autolab is internally connected to protective earth (P.E.). This can be an obstacle when measurement is desired of a cell that is itself in contact with P.E. In such a case, undefined currents will flow through the loop that is formed when the electrode connections from the PGSTAT are linked to the cell and measurements will not be possible. Please note that not only a short circuit or a resistance can make a connection to earth, but also a capacitance is capable of providing a conductive path (for AC signals). The earth connection between the cell and P.E. should always be broken. If there is no possibility of doing this, please contact Metrohm Autolab for a custom solution, if available.

4.3.13 – Environmental conditions

The PGSTAT may be used at temperatures of 0 to 40 degrees Celsius. The instrument is calibrated at 25 degrees Celsius and will show minimum errors at that temperature. The ventilation holes on the bottom plate and on the rear panel may never be obstructed, nor should the instrument be placed in direct sunlight or near other sources of heat.

4.3.14 – Temperature overload

As a safety precaution, the PGSTAT is equipped with a circuit that monitors the temperature of the internal power electronics. A temperature overload will be displayed as a blinking indicator in the manual cell switch, with the cell

automatically turned off. You will not be able to turn the cell back on until the temperature inside the instrument has fallen to an acceptable level. It can then be switched on again by pressing the manual cell switch button on the front panel.

During normal operation the temperature should never become extremely high and no temperature overload will occur. If this does happen, the origin of the temperature overload should be identified:

1. Is the room temperature unusually high?
2. Was the PGSTAT oscillating?
3. Is the voltage selector for mains power set to the right value?
4. Is the fan turning and are all the ventilation holes unobstructed?
5. Was the cell delivering a considerable amount of power to the PGSTAT?
6. Are the WE and CE cables shorted in PSTAT mode[48]?

If a temperature overload takes place repeatedly, for no obvious reason, Metrohm Autolab recommends having the instrument checked by their service department.

4.3.15 – Noise

When measuring low level currents, some precautions should be taken in order to minimize noise. The personal computer must be placed as far away as possible from the electrochemical cell and the cell cables. The cell cables should not cross other electrical cables. Other equipment with power supplies can also cause noise. For instance, the interface for mercury electrodes IME should also be placed with some care. If possible place the computer between the PGSTAT and other equipments. Avoid using unshielded extension cables to the electrodes. The use of a Faraday cage is also advised.

If the cell system has a ground connector, it can be connected to the analog ground connector at the front of the PGSTAT. If a Faraday cage is used, it should be connected to this ground connector. Some experiments concerning optimization of the signal-to-noise ratio can readily indicate whether or not a configuration is satisfactory.

More information on noise is provided in section 4.8.

[48] This must never occur!

4.4 – Autolab PGSTAT302F information

This section provides specific information for the Autolab PGSTAT302F. The PGSTAT302F is a special version of the Autolab PGSTAT302N which can be operated in so-called **floating** mode[49]. In floating mode, the PGSTAT302F can be used to control the potential of grounded working electrodes. In this configuration, the Autolab is floating with respect to the working electrode sample. Additionally, the PGSTAT302F can be operated in non-floating mode in combination with working electrode disconnected from ground.

Warning

The floating mode of the special PGSTAT302N must only be used on grounded working electrodes. The working electrode can be grounded using the green ground connector embedded in the CE/WE cable of the PGSTAT302F.

Warning

Instrument performance can be substantially degraded when the PGSTAT302F is operated in floating mode. The instrument specifications provided by Metrohm Autolab can only be achieved when the PGSTAT302F is used in non-floating mode.

Note

Special precautions must be taken with the cell connections when the PGSTAT302F is used in floating mode. Only the working electrode can be connected to ground, all other electrodes must be isolated from ground. External equipments connected to the PGSTAT302F must be isolated when the instrument is used in floating mode. Keep in mind that grounding of external equipment can occur through connections to a computer, if applicable (for example through a USB or RS232 cable).

[49] The compliance voltage of the PGSTAT302F is +/- 10 V in floating mode. The compliance voltage in grounded mode is +/- 10 V with the default cell cables and +/- 30 V with optional modified cell cables. Please contact your Autolab distributor for more information.

4.4.1 – Front panel and cell cable connection

There are four connectors on the front panel of the PGSTAT. The cable that connects to the WE and CE should be plugged into the WE/CE socket while the cable with the differential amplifier (leading to the RE, S and optionally WE2 electrodes) connects to the RE/S socket. A ground cable, embedded in the WE/CE cable connection, can be used to plug to the earth bulkhead for shielding purposes. Finally a monitor cable can be connected to a dedicated connector (see Figure 4.16).

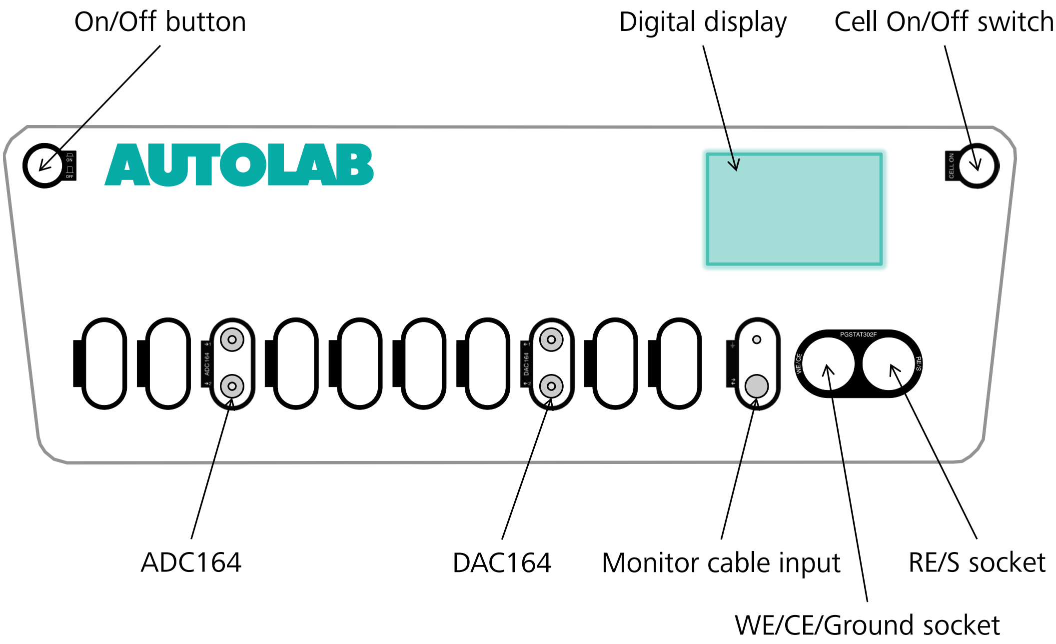


Figure 4.16 – Overview of the Autolab PGSTAT302F

The cell cables are labelled as follows:

- Working or indicator electrode, WE (red)
- Sense electrode, S (red)
- Reference electrode, RE (blue)
- Auxiliary or counter electrode, CE (black)

In a four electrode setup, each of the cell cable connectors is used independently. In a three electrode set-up the working electrode and sense lead are both connected to the working electrode. In a two electrode set-up the counter and reference electrode lead are both connected to the same electrode (see Figure 4.17).

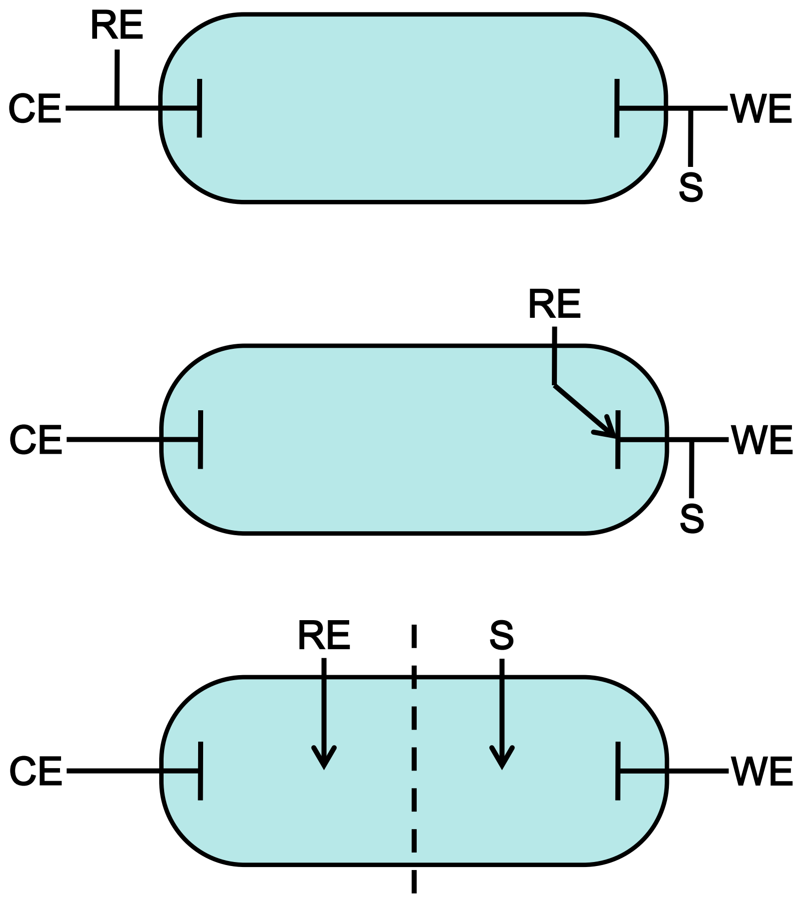


Figure 4.17 – Overview of the possible cell connections with the Autolab PGSTAT302F (from top to bottom: two electrode, three electrode and four electrode setup)

4.4.2 – Power up

The settings of the PGSTAT on power-up are pre-defined. The following settings are used:

- Cell: off
- Mode: Potentiostatic
- Bandwidth: High stability
- iR Compensation: off
- Current range: 10 mA

Warning

In floating mode, the i_{OVL} warning may be lit when the cell is off. This warning can be ignored.

4.4.3 – Connections for analog signals

The Autolab PGSTAT302F provides connections for analog signals through two different types of connectors:

- BNC connectors directly located on the front panel of the instrument
- BNC connectors located on the monitor cable

4.4.3.1 – Connections for analog signals (front panel)

The ADC164 module and the DAC164 module are fitted with two analog inputs and two analog outputs, respectively (see Figure 4.18).

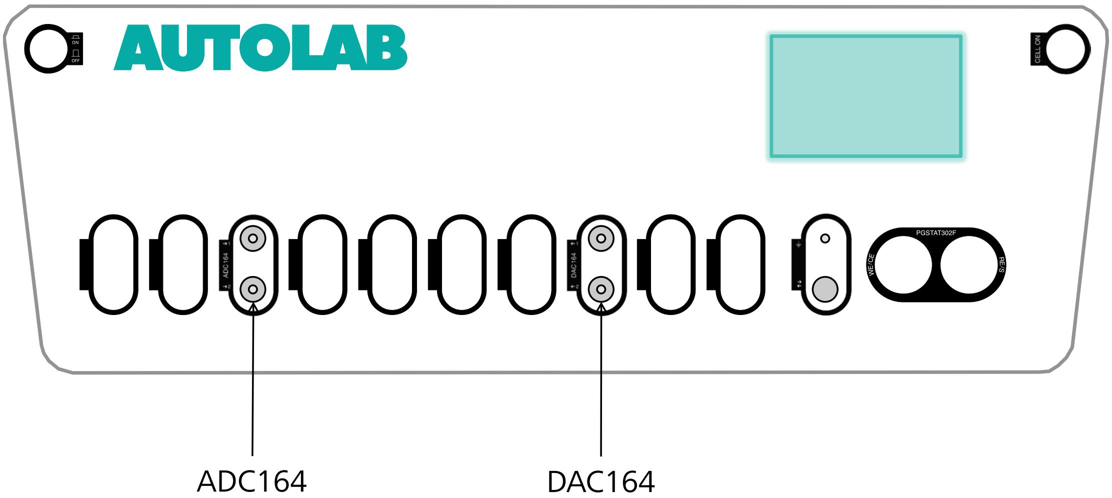


Figure 4.18 – Overview of the connections for analog signals provided on the front panel of the Autolab PGSTAT (ADC164 and DAC164)

ADC164 – The ADC164 inputs, labelled →1 and →2 on the front panel, can be used to record any analog signal with a ± 10 V value range. The input impedance of the two analog inputs is 50 Ω.

DAC164 – The DAC164 outputs, labelled ←1 and ←2 on the front panel, can be used to generate any analog signal with a ± 10 V value range. The output impedance of these two inputs is 50 Ω. Corrections should be made with loads < 100 kΩ. Because of dissipation, the minimum load impedance should be 200 Ω.

Note

The DAC164 ←1 and ←2 outputs are identified as DAC channels 3 and 4, respectively in the *Set DAC* command.

These inputs are floating when the PGSTAT302F is operated in floating mode. Connected equipment may <u>not</u> be connected to ground and the shield of the BNC cables may <u>not</u> be connected to safety ground.

Avoid creating ground loops as this will often degrade the performance of the PGSTAT302F.

4.4.3.2 – Connections for analog signals (monitor cable)

With the supplied monitor cable, there are a number of BNC connectors to the PGSTAT analog circuits (see Figure 4.19). All the signals are with respect to Autolab ground and indirectly to protective earth when the PGSTAT302F is operated in normal mode.

These signals are floating when the PGSTAT302F is operated in floating mode. Connected equipment may **not** be connected to ground and the shield of the BNC cables may **not** be connected to safety ground.

Avoid creating ground loops as this will often degrade the performance of the PGSTAT302F.

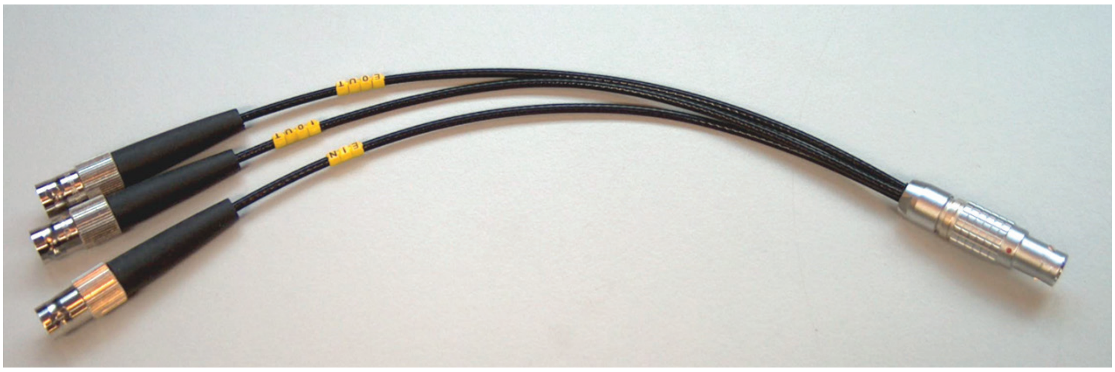


Figure 4.19 – The monitor cable for the PGSTAT302F

The following signals are available:

E_{OUT} – This output corresponds to the differential potential of S versus RE[50]. The output voltage will vary between ±10 V. The output impedance is 50 Ω, so a correction should be made if a load < 100 kΩ is connected. The minimum load impedance is 200 Ω.

i_{OUT} – This signal corresponds to the output of the current-to-voltage converter circuit of the PGSTAT[51]. A 1 V signal corresponds to -{1 x the selected current range}. The output level varies between ± 10 V. The output impedance is 50 Ω, so a correction should be made if a load < 100 kΩ is connected. The minimum load impedance is 200 Ω.

E_{IN} – This is an analog voltage input, that can only be used after it has been enabled in software, using the *Autolab control* command (see Figure 4.20). Do not leave it enabled unnecessarily, to prevent noise pickup by the system. This input is directly connected to the summation point, Σ, of the PGSTAT. In PSTAT mode, a

[50] The E_{out} value corresponds to WE(1).Potential.
[51] The i_{out} value corresponds to -WE(1).Current.

1 V signal will add -1 V to the cell voltage, while in GSTAT mode a 1 V signal adds an extra current of -{1 x the selected current range} to flow. In both cases, the external signal adds to any pre-defined voltage or current. The input voltage range is ± 10 V. Input impedance is 1 kΩ (only when input is activated) so a correction should be made when the source impedance is > 1 Ω.

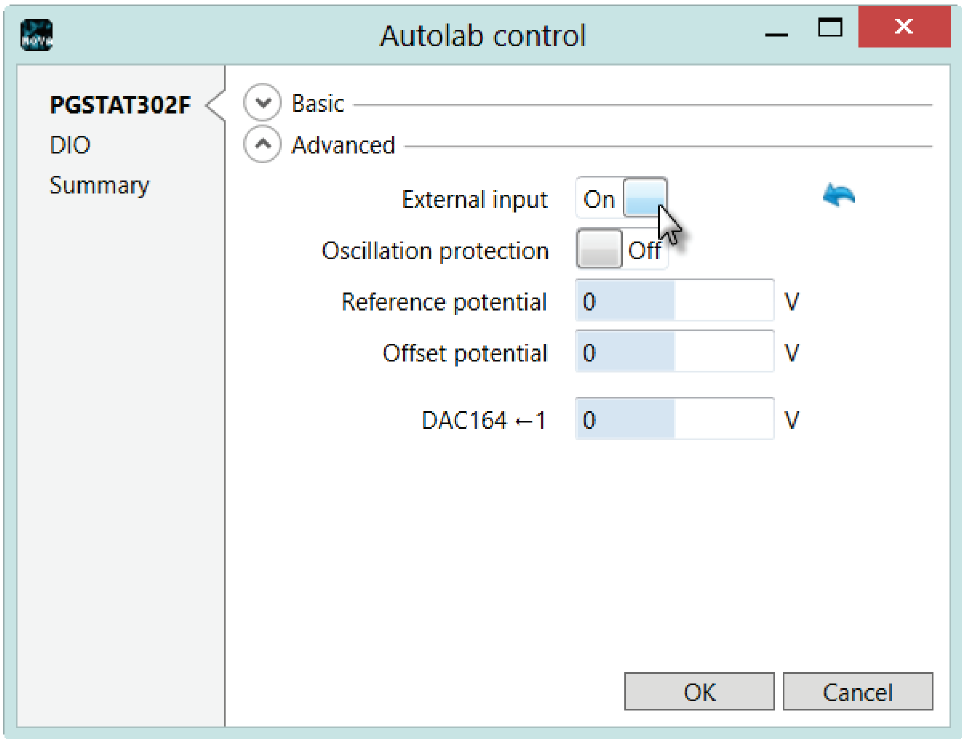


Figure 4.20 – The external input is enabled in the Autolab control window

4.4.4 – High stability and High speed

The PGSTAT302F is equipped with two different bandwidth settings: High stability (HSTAB) and High speed. The bandwidth can be defined using the Autolab control command (see Figure 4.21).

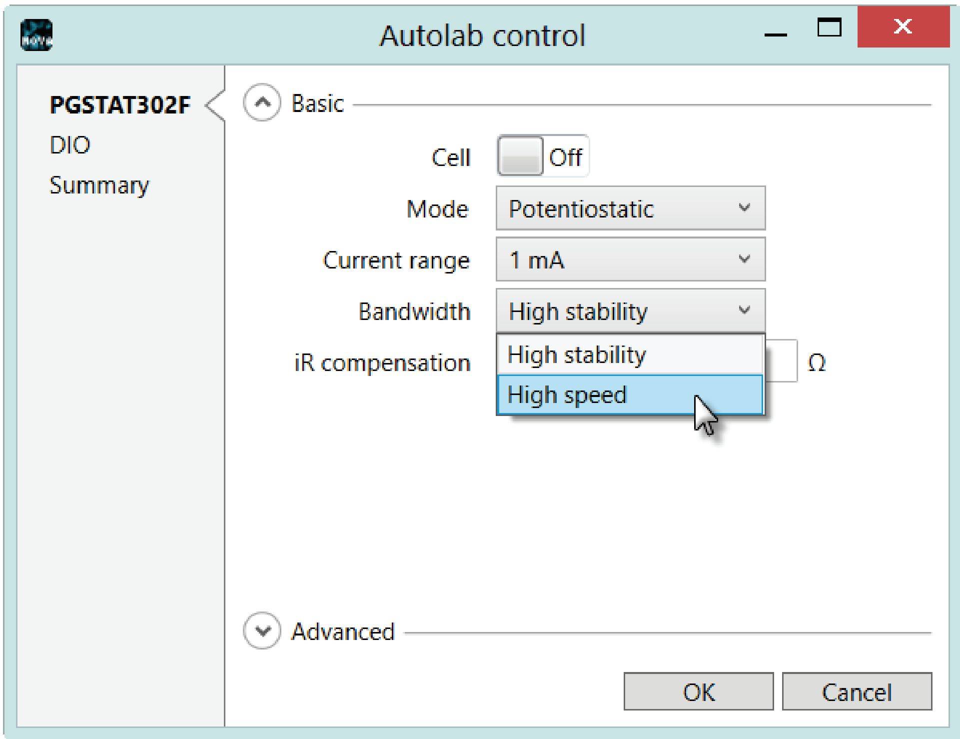


Figure 4.21 – The Autolab control window can be used to set the bandwidth of the PGSTAT

The purpose of these different modes of operation is to provide a maximum bandwidth, maintaining stability in the PSTAT or GSTAT control loop. The normal mode of operation is High stability[52]. This gives the Control Amplifier a bandwidth of 12.5 kHz. The HSTAB indicator on the front panel of the PGSTAT and in the Autolab display is lit when the High stability mode is active (see Figure 4.22).

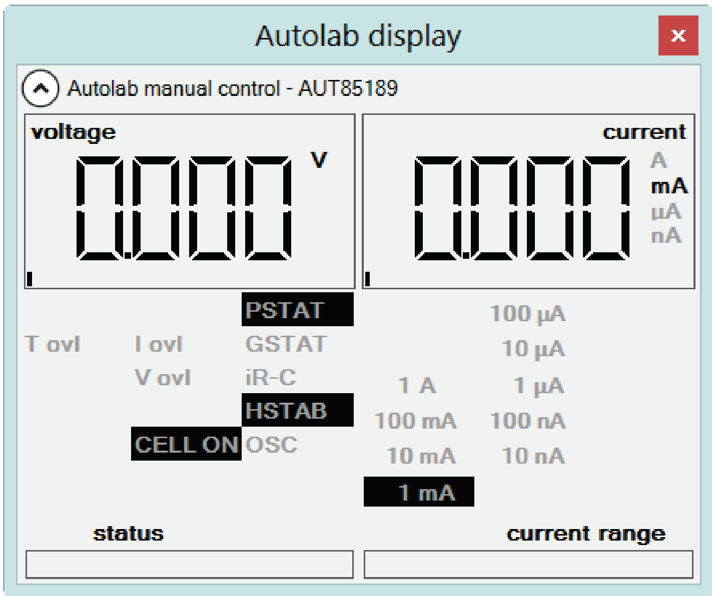


Figure 4.22 – A HSTAB indicator is provided on the Autolab display

[52] Power up default setting.

This setting is the most appropriate for measurements at low frequencies or low scan rates. The noise in the i and E signals will be minimized. Measurements at high frequency or at high scan rates require a faster mode of operation.

When operating in High speed mode, the control amplifier will have its bandwidth extended with one decade up to 125 kHz. Some cells can show ringing or oscillation using this setting, particularly highly capacitive cells in PSTAT mode. Increasing the bandwidth also increases the noise levels for the i and E signals. The High speed mode is automatically selected during impedance measurement at frequencies > 10 kHz.

Note

It is possible to switch from High stability to High speed by clicking the HSTAB label in the Autolab display. In High speed mode, this label will be unlit, both on the front panel of the PGSTAT and on the Autolab display. Clicking the HSTAB label again switches the bandwidth back to High stability.

The High speed mode is automatically selected during impedance measurements at frequencies > 10 kHz, while the High stability mode is selected for frequencies below 10 kHz (see Figure 4.23).

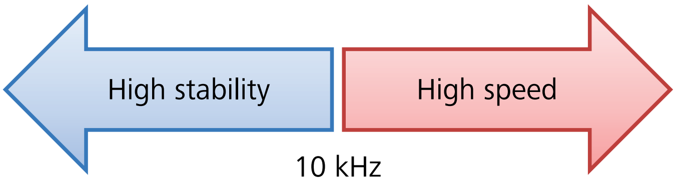


Figure 4.23 – Bandwidth limits in the Autolab PGSTAT302F

Warning

The higher the bandwidth, the more important it is to pay attention to adequate shielding of the cell and the electrode connectors. The use of a Faraday cage is recommended in this case.

4.4.5 – RE input impedance and stability

The electrometer RE input contains a small capacitive load. If the capacitive part of the impedance between CE and RE is comparatively large, phase shifts will occur which can lead to instability problems when working in potentiostatic mode. If the impedance between the CE and the RE cannot be changed and oscillations are observed, it is recommended to select the High stability mode to increase the system stability. In general, the use of High stability leads to a more stable control loop, compared to High speed or Ultra high speed and a significantly lower bandwidth.

To make use of the full potentiostat bandwidth (High speed mode), the impedance between CE and RE has to be lower than 35 kΩ[53]. This value is derived by testing. In galvanostat mode, this large impedance between CE and RE, will usually not lead to stability problems, because of the current feedback regulation.

4.4.6 – Galvanostatic FRA measurements

The capacitive part of the impedance between RE and ground is an important aspect to consider when performing FRA measurements in galvanostat mode. Large reference electrode impedance values may introduce a phase shift at low frequencies. The origin of the phase shift between the CE and the RE cannot be determined from the FRA data.

Galvanostatic FRA measurements at 1 MHz require a maximum of 3 kΩ reference electrode impedance to keep phase errors within the ± 5 ° limit.

4.4.7 – Galvanostat, potentiostat and iR-compensation bandwidth

For galvanostatic measurements on low current ranges, the bandwidth limiting factor becomes the current-to-voltage circuit rather than the control amplifier.

For stability reasons it is not recommended to use the High speed mode for current ranges < 10 µA.

As the current measurement circuit plays an important role in the iR compensation technique, its use is also subject to bandwidth limitations. A general indication of the maximum available bandwidth for GSTAT and for iR compensation can be found in Table 4.4:

Mode	GSTAT	iR/C - PSTAT
1 A – 1 mA	> 500 kHz	> 500 kHz
100 µA	125 kHz	500 kHz
10 µA	100 kHz	100 kHz
1 µA	10 kHz	10 kHz
100 nA	1 kHz	1 kHz
10 nA	100 Hz	100 Hz

Table 4.4 – Bandwidth overview for the PGSTAT302F

At the same time, the iR-compensation bandwidth limits indicate up to which frequency current measurements can be made in potentiostatic mode (either with or without iR compensation).

[53] Empirical value.

4.4.8 – Galvanostatic operation and current range linearity

For galvanostatic experiments, automatic current ranging is not possible. The measurements are performed in a fixed current range. Each current range on the instrument is characterized by a specific linearity limit and this specification determines the maximum current that can be applied in galvanostatic mode.

The linearity limitation also applies on measurements performed in potentiostatic mode in a fixed current range.

Table 4.5 provides an overview of the current range linearity for the PGSTAT302F.

Current range	Linearity
1 A	2
100 mA	3
10 – 1 mA	3
100 – 1 µA	3
100 – 10 nA	3

Table 4.5 – Linearity limit for the PGSTAT302F

For example, in the 100 mA current range, the maximum current that can be applied, galvanostatically, using the PGSTAT302F, is 300 mA. The maximum current that can be measured in the 100 mA current range, using the same instrument is 1000 mA, although currents exceeding 300 mA will be measured outside of the linearity limit of this current range.

In galvanostatic operation, the applied current values are checked during the procedure validation step. When the applied current exceeds the linearity limit for the specified current range, an error message will be shown in the procedure validation screen (see Figure 4.24).

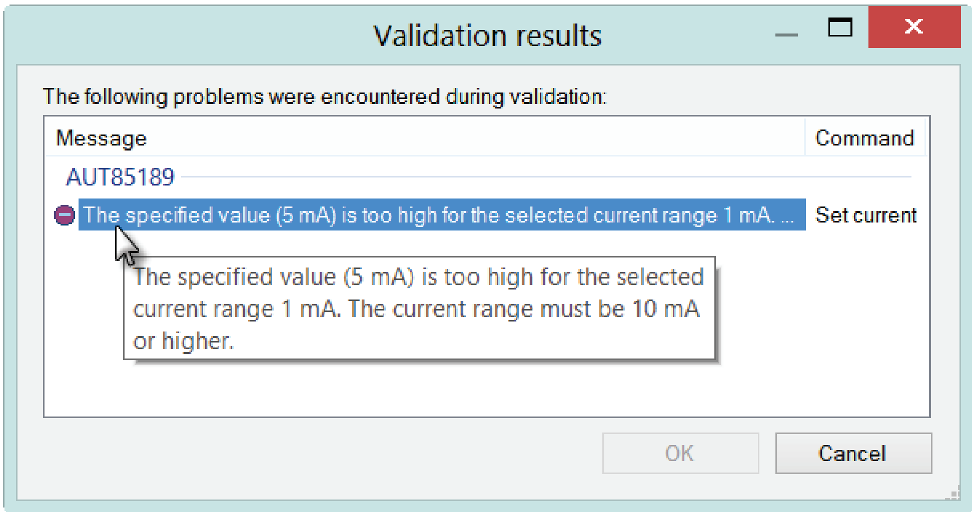


Figure 4.24 – The procedure validation step always checks the applied current values for the allowed linearity

Note

In potentiostatic mode, this check is not performed. It is possible to measure a current value in a fixed current range, even if the current value exceeds the linearity limit of the active current range. This triggers a current overload warning. When this happens during a measurement, a message will be shown in the user log, suggesting a modification of the current range (see Figure 4.25).

User log message	Time	Date	Command
Autolab/USB connected (AUT85189)	4:05:04 PM	1/15/2013	-
Overload occurred in 1 µA current range, use a higher current range.	4:10:43 PM	1/15/2013	CV staircase

Figure 4.25 – When a current overload is detected, a suggestion is shown in the user log

4.4.9 – Oscillation detection

The PGSTAT302F has a detector for large-amplitude oscillation. The detector will spot any signal swing that causes the control amplifier to produce both a positive and a negative Voltage overload within ~ 200 µs. Thus, large oscillations at frequencies > 2.5 kHz will be detected. Upon oscillation, the OSC indicator on the PGSTAT front panel will be activated. The V_{ovl} warning will also be shown in the Autolab display. An oscillation protection feature can be enabled or disabled in the software, using the *Autolab control* command (see Figure 4.26).

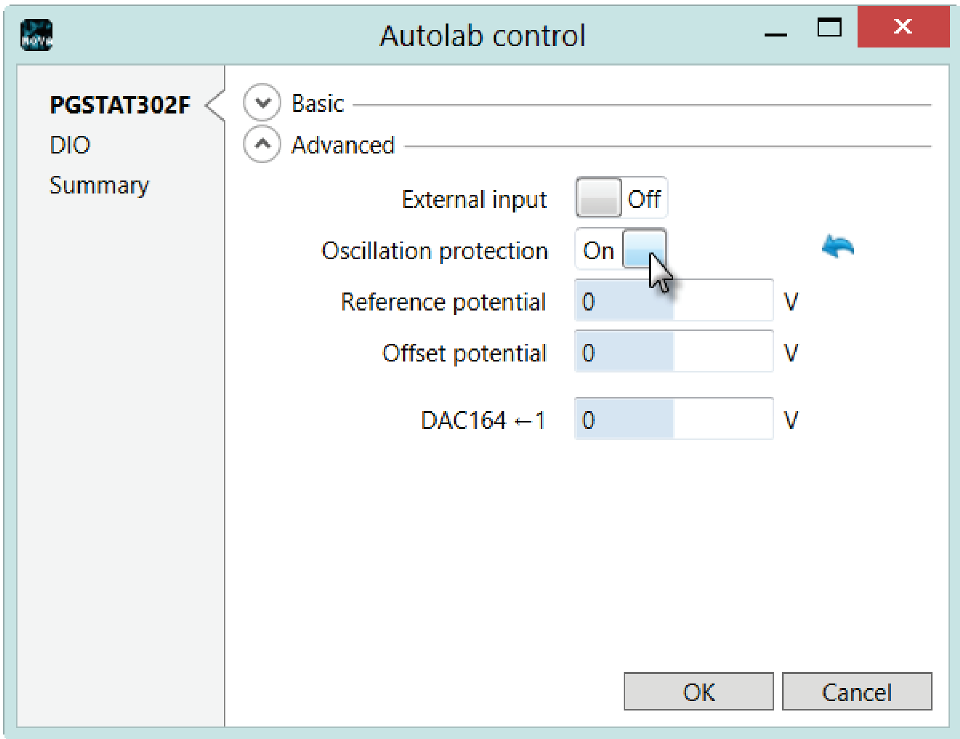


Figure 4.26 – The Autolab control window can be used to switch the oscillation protection on or off

If the oscillation protection is enabled, the occurrence of oscillation will also immediately turn off the manual cell switch of the Autolab. When this happens, both the OSC indicator and the manual cell switch start blinking. The Autolab display will show the message 'Cell manually off' (see Figure 4.27).

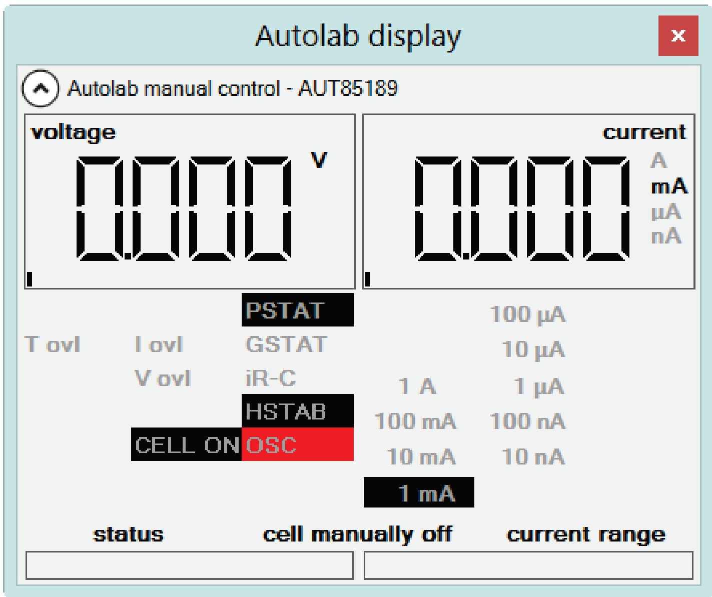


Figure 4.27 – The cell manually off is displayed when the oscillation protection circuit is triggered

The cell may be switched on again by pressing the manual cell switch button. If oscillation resumes, the cell switch will be turned off as soon as the button is released. Holding the button pressed in, provides an opportunity to observe the system during oscillation.

Some cells that cause ringing when switching the cell on or changing the current range can falsely trigger the oscillation detector. If this happens, the Oscillation protection may be switched off in the software in order to prevent an accidental disconnection of the cell.

4.4.10 – Maximum reference electrode voltage

The differential electrometer input contains an input protection circuitry that becomes active after crossing the ±10 V limit. This is implemented to avoid electrometer damage. Please note that the V_{ovl} indicator will not light up for this type of voltage overload. The measured voltage will be cutoff at an absolute value of 10.00 V.

Depending on the cell properties, galvanostatic control of the cell could lead to a potential difference between the RE and the S/WE larger than 10 V. This situation will trigger the cutoff of the measured voltage to prevent overloading the differential amplifier.

4.4.11 – Active cells

Some electrochemical cells such as batteries and fuel cells are capable of delivering power to the PGSTAT302F. This is allowed only to a maximum 'cell' power, P_{MAX} of 20 W.

This means that cells showing an absolute voltage ($|V_{cell}|$) of less than 10 V between WE and CE are intrinsically safe. They may drive the PGSTAT output stage into current limit but will not overload the amplifier. On the other hand, cells that have an absolute voltage higher than 10 V between WE and CE may only deliver a maximum current, i_{MAX} given by:

$$i_{MAX} = \frac{P_{MAX}}{|V_{MAX}|}$$

4.4.12 – Grounded cells and grounded working electrodes

The PGSTAT302F can be operated in two different modes:

- **Normal mode:** this mode corresponds to the operating mode using in all the PGSTAT instruments. For more information on the restrictions applying to this mode, please refer to section 4.3.12.
- **Floating mode:** this mode is only available on the PGSTAT302F. In this mode, measurement circuitry of the Autolab is internally disconnected to protective earth (P.E.). This allows the instrument to be used in combination with a grounded working electrode or a grounded cell.

The PGSTAT302F can be set to either **normal** mode or **floating** mode using a dedicated short-circuit plug on the back plane of the instrument (see Figure 4.28). When the short-circuit plug is connected as shown in Figure 4.28, the instrument operates in **normal** mode. When the short-circuit plug is disconnected from the back panel, the instrument operates in **floating** mode.

Figure 4.28 – The PGSTAT302F can be set to normal mode (left) or to floating mode (right) using the provided short-circuit plug

4.4.13 – Environmental conditions

The PGSTAT302F may be used at temperatures of 0 to 40 degrees Celsius. The instrument is calibrated at 25 degrees Celsius and will show minimum errors at that temperature. The ventilation holes on the bottom plate and on the rear panel may never be obstructed, nor should the instrument be placed in direct sunlight or near other sources of heat.

4.4.14 – Temperature overload

As a safety precaution, the PGSTAT302F is equipped with a circuit that monitors the temperature of the internal power electronics. A temperature overload will be displayed as a blinking indicator in the manual cell switch, with the cell automatically turned off. You will not be able to turn the cell back on until the temperature inside the instrument has fallen to an acceptable level. It can then be switched on again by pressing the manual cell switch button on the front panel.

During normal operation the temperature should never become extremely high and no temperature overload will occur. If this does happen, the origin of the temperature overload should be identified:

1. Is the room temperature unusually high?
2. Was the PGSTAT oscillating?
3. Is the voltage selector for mains power set to the right value?
4. Is the fan turning and are all the ventilation holes unobstructed?
5. Was the cell delivering a considerable amount of power to the PGSTAT?
6. Are the WE and CE cables shorted in PSTAT mode[54]?

If a temperature overload takes place repeatedly, for no obvious reason, Metrohm Autolab recommends having the instrument checked by their service department.

4.4.15 – Noise

When measuring low level currents, some precautions should be taken in order to minimize noise. The personal computer must be placed as far away as possible from the electrochemical cell and the cell cables. The cell cables should not cross other electrical cables. Other equipment with power supplies can also cause noise. For instance, the interface for mercury electrodes IME should also be placed with some care. If possible place the computer between the PGSTAT302F and other equipments. Avoid using unshielded extension cables to the electrodes. The use of a Faraday cage is also advised.

[54] This must never occur!

Warning

Instrument performance can be substantially degraded when the PGSTAT302F is operated in floating mode. The instrument specifications provided by Metrohm Autolab can only be achieved when the PGSTAT302F is used in non-floating mode.

If the cell system has a ground connector, it can be connected to the analog ground connector at the front of the PGSTAT302F. If a Faraday cage is used, it should be connected to this ground connector. Some experiments concerning optimization of the signal-to-noise ratio can readily indicate whether or not a configuration is satisfactory.

More information on noise is provided in Section 4.8.

4.5 – Autolab PGSTAT101 and M101 information

This section provides specific information for the Autolab PGSTAT101 and the M101 potentiostat/galvanostat module for the Multi Autolab.

4.5.1 – Front panel and cell cable connection (PGSTAT101)

There are two connectors on the front panel of the PGSTAT101. The cell cable should be plugged into the CELL socket on the front panel of the instrument. The I/O socket on the front panel can be used to connect the optional I/O cable (see Figure 4.29).

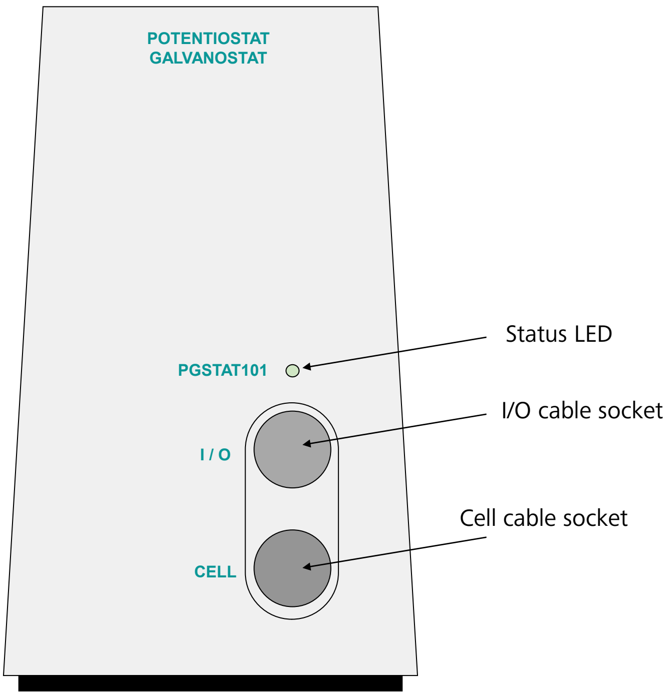


Figure 4.29 – Overview of the Autolab PGSTAT101 (front)

The cell cable is labelled as follows:

- Working or indicator electrode, WE (red)
- Sense electrode, S (red)
- Reference electrode, RE (blue)
- Auxiliary or counter electrode, CE (black)

An additional ground connection (for shielding purposes, e.g. a Faraday cage) is also provided with the cell cable.

In a four electrode setup, each of the cell cable connectors is used independently. In a three electrode set-up the working electrode and sense lead are both connected to the working electrode. In a two electrode set-up the counter and reference electrode lead are both connected to the same electrode (see Figure 4.30).

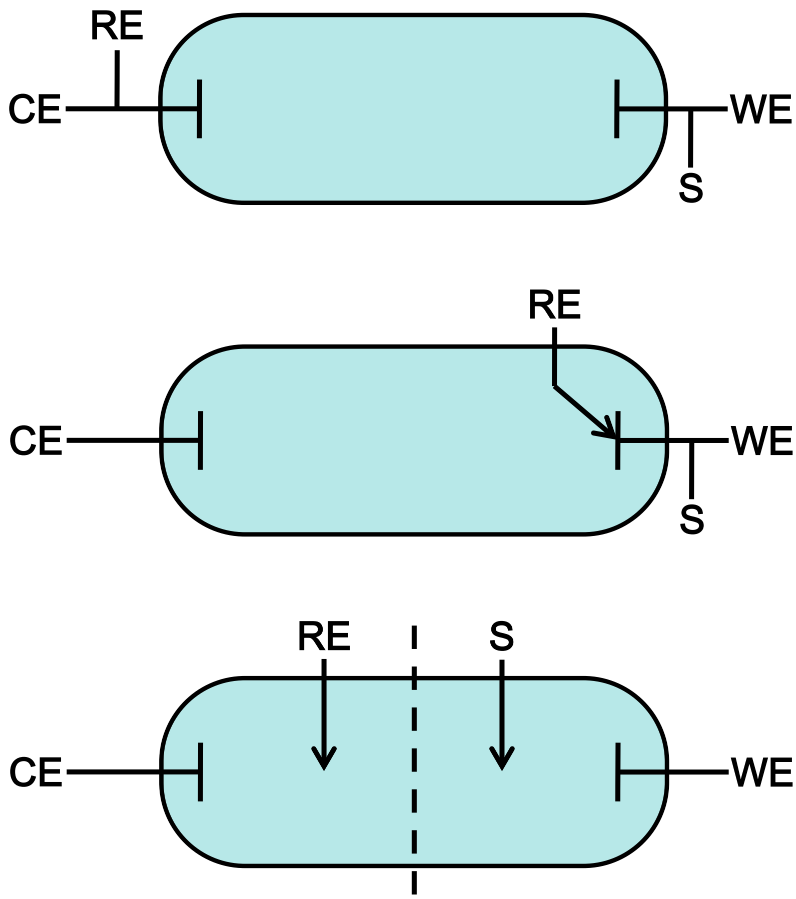


Figure 4.30 – Overview of the possible cell connections with the Autolab PGSTAT101 (from top to bottom: two electrode, three electrode and four electrode setup)

4.5.2 – Front panel and cell cable connection (M101)

The M101 potentiostat/galvanostat module can be installed in the Multi Autolab frame, up to a maximum of 12 modules in a single frame. The M101 is identical in

specification to the PGSTAT101. All information provided in the rest of section 4.5 applies to both the PGSTAT101 and the M101[55] installed in the Multi Autolab.

There are three connectors on the front panel of each M101 module installed in the Multi Autolab (see Figure 4.31).

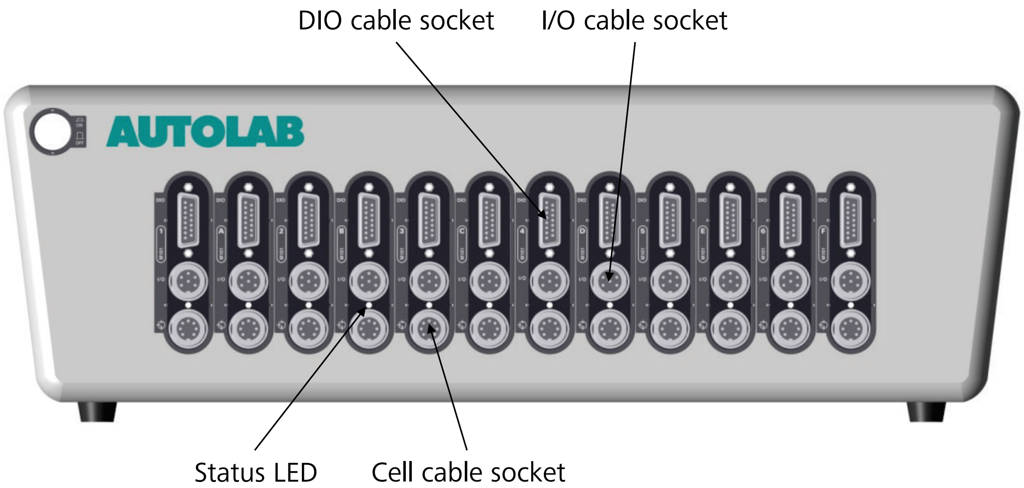


Figure 4.31 – Overview of the M101 (front) in Multi Autolab frame

Each M101 module is identified by a module label on the front panel, indicating the location and the purpose of each connector (see Figure 4.32)

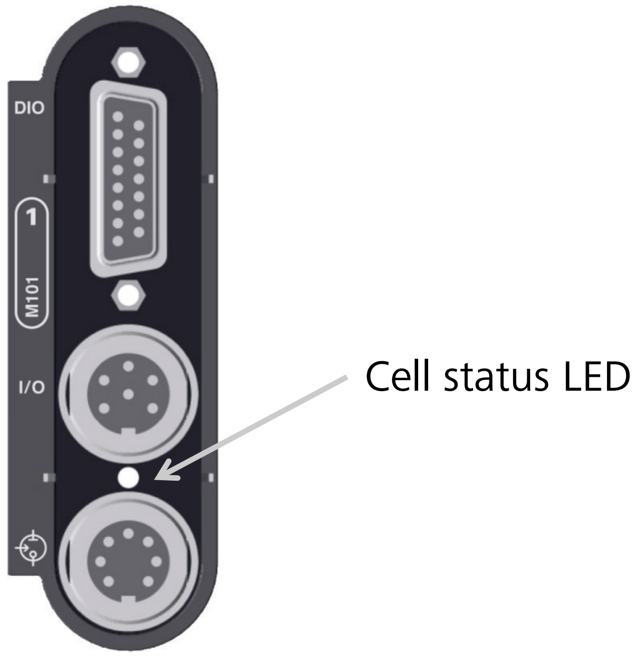


Figure 4.32 – Detailed view of the M101 module label

[55] In the rest of this section, the M101 and PGSTAT101 will be referred to as PGSTAT101.

The cell cable should be plugged into the lowest socket, labelled by the symbol ⌖, on the front panel of the module. The I/O socket on the front panel can be used to connect the optional I/O cable. The DIO cable, used to connect to the optional IME663 or IME303 or for TTL triggering, can be connected to the DIO connector on the front panel.

Note

Specific information on Multi Autolab can be found in the Multi Autolab tutorial, available from the Help menu.

The cell cable is labelled as follows:

- Working or indicator electrode, WE (red)
- Sense electrode, S (red)
- Reference electrode, RE (blue)
- Auxiliary or counter electrode, CE (black)

An additional ground connection (for shielding purposes, e.g. a Faraday cage) is also provided with the cell cable.

In a four electrode setup, each of the cell cable connectors is used independently. In a three electrode set-up the working electrode and sense lead are both connected to the working electrode. In a two electrode set-up the counter and reference electrode lead are both connected to the same electrode (see Figure 4.30).

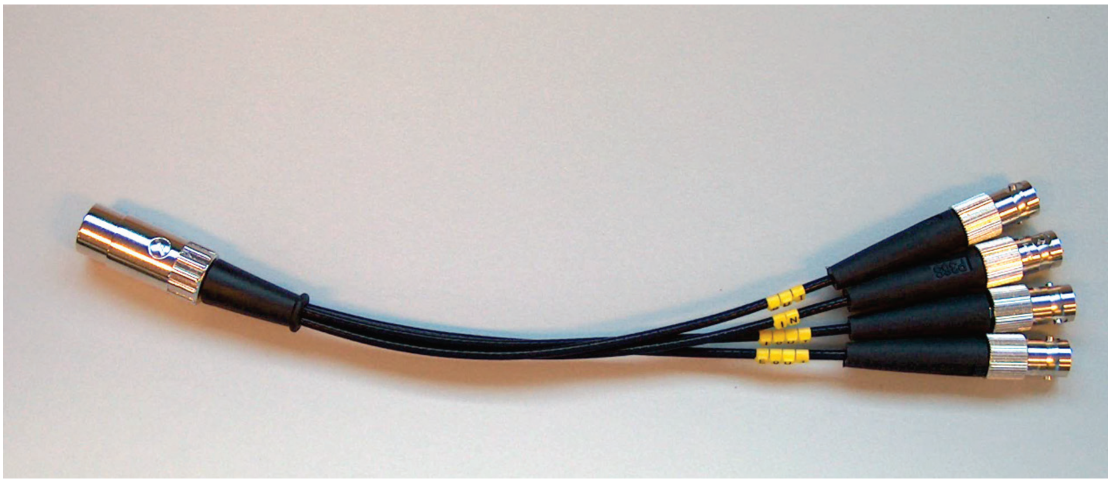
4.5.3 – Power up

The settings of the PGSTAT101 on power-up are pre-defined. The following settings are used:

- Cell: off
- Mode: Potentiostatic
- Bandwidth: High stability
- iR Compensation: off
- Current range: 1 µA

4.5.4 – Connections for analog signals

With the optional I/O cable, four additional connections are provided to the PGSTAT101 analog circuits (see Figure 4.33). All the signals are with respect to Autolab ground and indirectly to protective earth. Avoid creating ground loops as this will often degrade the performance of the PGSTAT.



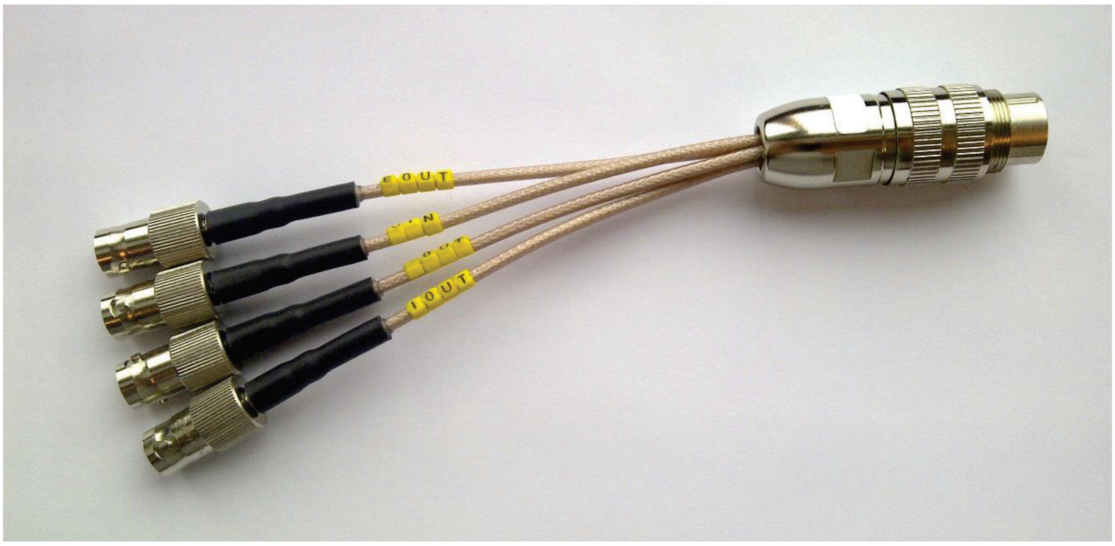


Figure 4.33 – The optional I/O cable for the PGSTAT101 (top) and M101 (bottom)

The following signals are available:

E_{OUT} – This output corresponds to the differential potential of RE versus S[56]. The output voltage will vary between ±10 V. The output impedance is 1 kΩ, so a correction should be made if a load < 2 MΩ is connected. The maximum bandwidth is 300 kHz.

i_{OUT} – This signal corresponds to the inverted output of the current-to-voltage converter circuit of the PGSTAT101[57]. A 1 V signal corresponds to {-1 x the selected current range}. The output level varies between ± 10 V. The output impedance is 50 Ω, so a correction should be made if a load < 100 kΩ is connected. The minimum load impedance is 200 Ω.

V_{OUT} – This output corresponds to the DAC output. It is controlled by software and is meant to be used to control external devices, like the rotating speed of a

[56] The E_{out} value corresponds to -WE(1).Potential.
[57] The i_{out} value corresponds to -WE(1).Current/Current range.

Rotating Disc Electrode (RDE). The output level varies between ±10 V and the output impedance is very low, < 1 Ω. The output amplifier is capable of providing 5 mA at full scale, so load impedance should be > 2 kΩ.

V_{IN} – This input corresponds to the ADC input. This input can be used for measuring a second signal. The input range is ±10 V and the input impedance is 50 Ω.

4.5.5 – High stability, High speed and Ultra high speed

The PGSTAT101 is equipped with three different bandwidth settings: High stability (HSTAB), High speed and Ultra high speed. The bandwidth can be defined using the Autolab control command (see Figure 4.34).

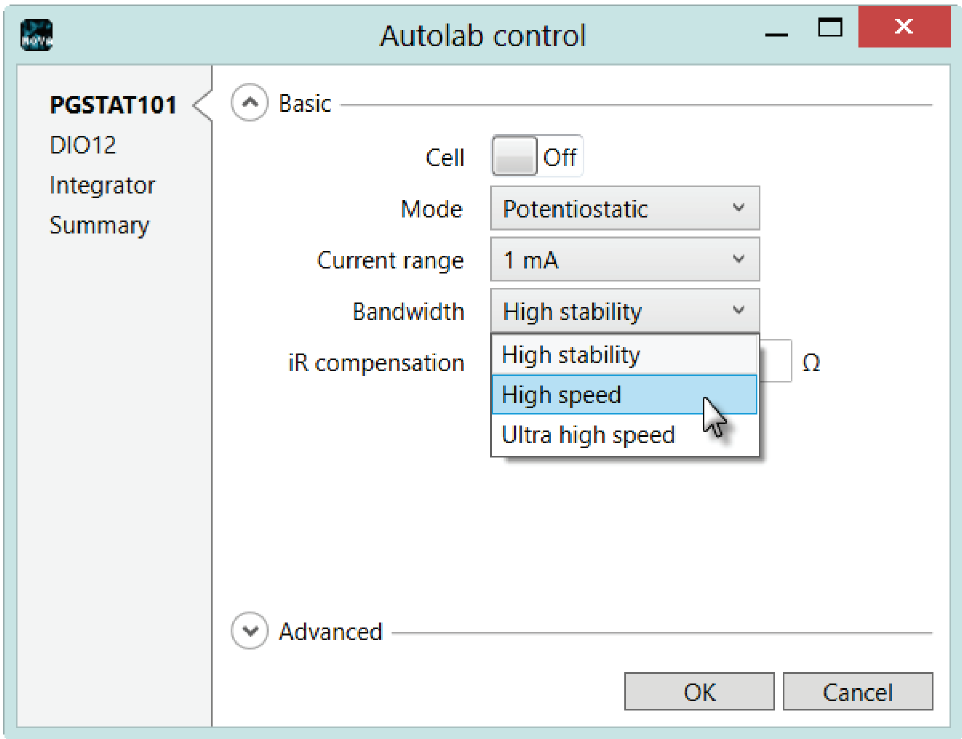


Figure 4.34 – The Autolab control window can be used to set the bandwidth of the PGSTAT101

The purpose of these different modes of operation is to provide a maximum bandwidth, maintaining stability in the PSTAT or GSTAT control loop. The normal mode of operation is High stability[58]. This gives the Control Amplifier a bandwidth of 12.5 kHz. The HSTAB indicator in the Autolab display is lit when the High stability mode is active (see Figure 4.35).

[58] Power up default setting.

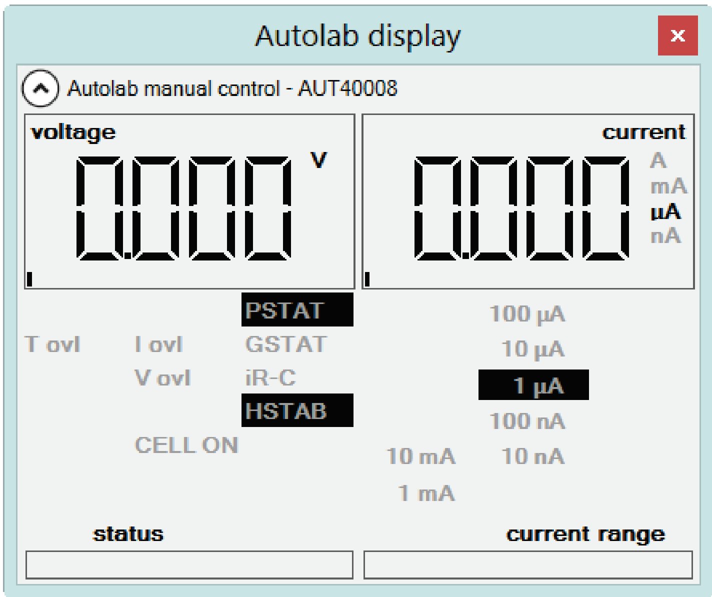


Figure 4.35 – A HSTAB indicator is provided on the Autolab display

This setting is the most appropriate for measurements at low frequencies or low scan rates. The noise in the i and E signals will be minimized. Measurements at high frequency or at high scan rates require a faster mode of operation.

When operating in High speed mode, the control amplifier will have its bandwidth extended with one decade up to 125 kHz. Some cells can show ringing or oscillation using this setting, particularly highly capacitive cells in PSTAT mode. Increasing the bandwidth also increases the noise levels for the i and E signals.

Note

It is possible to switch from High stability to High speed by clicking the HSTAB label in the Autolab display. In High speed mode, this label will be unlit on the Autolab display. Clicking the HSTAB label again switches the bandwidth back to High stability.

For applications requiring very high bandwidth, the Ultra high speed mode can be selected. In this mode, the control amplifier bandwidth is extended to 1 MHz. There is a significant oscillation risk using this setting, and the noise levels will generally show an increase relative to the High speed or High stability mode.

Warning

The higher the bandwidth, the more important it is to pay attention to adequate shielding of the cell and the electrode connectors. The use of a Faraday cage is recommended in this case.

4.5.6 – RE input impedance and stability

The electrometer RE input contains a small capacitive load. If the capacitive part of the impedance between CE and RE is comparatively large, phase shifts will occur which can lead to instability problems when working in potentiostatic mode. If the impedance between the CE and the RE cannot be changed and oscillations are observed, it is recommended to select the High stability mode to increase the system stability. In general, the use of High stability leads to a more stable control loop, compared to High speed or Ultra high speed and a significantly lower bandwidth.

To make use of the full potentiostat bandwidth (Ultra high speed mode), the impedance between CE and RE has to be lower than 35 kΩ[59]. This value is derived by testing. In galvanostat mode, this large impedance between CE and RE, will usually not lead to stability problems, because of the current feedback regulation.

4.5.7 – Galvanostat, potentiostat and iR-compensation bandwidth

For galvanostatic measurements on low current ranges, the bandwidth limiting factor becomes the current-to-voltage circuit rather than the control amplifier.

For stability reasons it is not recommended to use the High speed mode for current ranges < 10 µA. The Ultra high speed mode is also not recommended for current ranges < 1 mA.

As the current measurement circuit plays an important role in the iR compensation technique, its use is also subject to bandwidth limitations. A general indication of the maximum available bandwidth for GSTAT and for iR compensation can be found in Table 4.6:

Mode	GSTAT	iR/C - PSTAT
10 mA – 1 mA	> 1 MHz	> 1 MHz
100 µA	1 MHz	1 MHz
10 µA	10 kHz	75 kHz
1 µA	10 kHz	20 kHz
100 nA	400 Hz	4 kHz
10 nA	400 Hz	400 Hz

Table 4.6 – Bandwidth overview for the PGSTAT101

At the same time, the iR-compensation bandwidth limits indicate up to which frequency current measurements can be made in potentiostatic mode (either with or without iR compensation).

[59] Empirical value.

4.5.8 – Galvanostatic operation and current range linearity

For galvanostatic experiments, automatic current ranging is not possible. The measurements are performed in a fixed current range. Each current range on the instrument is characterized by a specific linearity limit and this specification determines the maximum current that can be applied in galvanostatic mode.

The linearity limitation also applies on measurements performed in potentiostatic mode in a fixed current range.

Table 4.7 provides an overview of the current range linearity for the PGSTAT101.

Current range	Linearity
10 mA	10
1 mA	7
10 – 1 mA	7
100 – 1 µA	7
100 – 10 nA	7

Table 4.7 – Linearity limit for the PGSTAT101

For example, in the 1 mA current range, the maximum current that can be applied, galvanostatically, using the PGSTAT101, is 7 mA. The maximum current that can be measured in the 1 mA current range is 10 mA, although currents exceeding 7 mA will be measured outside of the linearity limit of this current range.

In galvanostatic operation, the applied current values are checked during the procedure validation step. When the applied current exceeds the linearity limit for the specified current range, an error message will be shown in the procedure validation screen (see Figure 4.36).

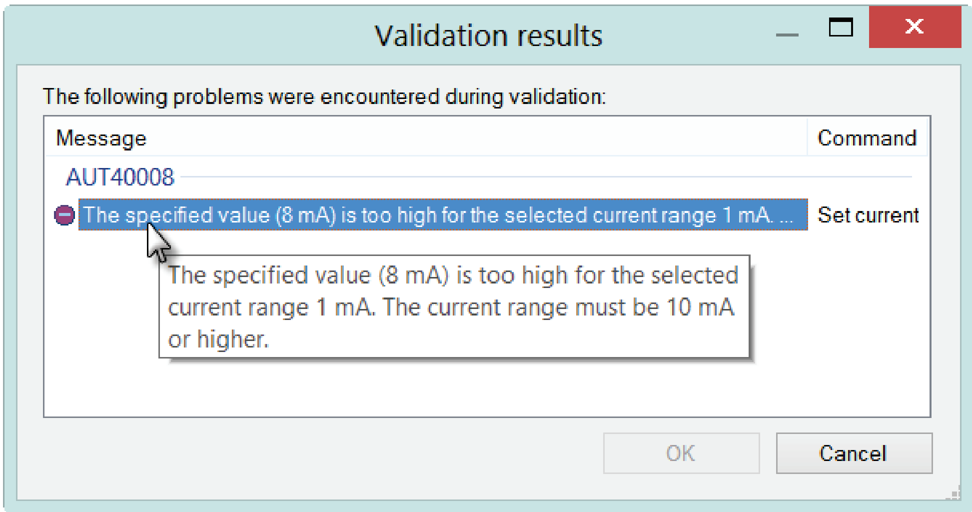


Figure 4.36 – The procedure validation step always checks the applied current values for the allowed linearity

Note

In potentiostatic mode, this check is not performed. It is possible to measure a current value in a fixed current range, even if the current value exceeds the linearity limit of the active current range. This triggers a current overload warning. When this happens during a measurement, a message will be shown in the user log, suggesting a modification of the current range (see Figure 4.37).

User log message	Time	Date	Command
Autolab/USB connected (AUT40008)	10:20:35 AM	1/15/2013	-
Overload occurred in 1 µA current range, use a higher current range.	10:23:18 AM	1/15/2013	CV staircase

Figure 4.37 – When a current overload is detected, a suggestion is shown in the user log

4.5.9 – Maximum reference electrode voltage

The differential electrometer input contains an input protection circuitry that becomes active after crossing the ±10 V limit. This is implemented to avoid electrometer damage. The red status LED indicator on the front panel not light up for this type of voltage overload. The measured voltage will be cutoff at an absolute value of 10.00 V.

Depending on the cell properties, galvanostatic control of the cell could lead to a potential difference between the RE and the S/WE larger than 10 V. This situation will trigger the cutoff of the measured voltage to prevent overloading the differential amplifier.

4.5.10 – Active cells

Some electrochemical cells such as batteries and fuel cells are capable of delivering power to the PGSTAT101. This is allowed only to a maximum ‘cell’ power, P_{MAX} of 8 W.

This means that cells showing an absolute voltage ($|V_{cell}|$) of less than 10 V between WE and CE are intrinsically safe. They may drive the PGSTAT101 output stage into current limit but will not overload the amplifier. On the other hand, cells that have an absolute voltage higher than 10 V between WE and CE may only deliver a maximum current, i_{MAX} given by:

$$i_{MAX} = \frac{P_{MAX}}{|V_{MAX}|}$$

4.5.11 – Grounded cells

The measurement circuitry of the Autolab is internally connected to protective earth (P.E.). This can be an obstacle when measurement is desired of a cell that is itself in contact with P.E. In such a case, undefined currents will flow through the loop that is formed when the electrode connections from the PGSTAT101 are linked to the cell and measurements will not be possible. Please note that not only a short circuit or a resistance can make a connection to earth, but also a capacitance is capable of providing a conductive path (for AC signals). The earth connection between the cell and P.E. should always be broken. If there is no possibility of doing this, please contact Metrohm Autolab for a custom solution, if available.

4.5.12 – Environmental conditions

The PGSTAT101 may be used at temperatures of 0 to 40 degrees Celsius. The instrument is calibrated at 25 degrees Celsius and will show minimum errors at that temperature. The ventilation holes on the bottom plate and on the rear panel may never be obstructed, nor should the instrument be placed in direct sunlight or near other sources of heat.

4.5.13 – Noise

When measuring low level currents, some precautions should be taken in order to minimize noise. The personal computer must be placed as far away as possible from the electrochemical cell and the cell cables. The cell cables should not cross other electrical cables. Other equipment with power supplies can also cause noise. For instance, the interface for mercury electrodes IME should also be placed with some care. If possible place the computer between the PGSTAT101 and other equipments. Avoid using unshielded extension cables to the electrodes. The use of a Faraday cage is also advised.

If the cell system has a ground connector, it can be connected to the analog ground connector provided with the cell cable of the PGSTAT101. If a Faraday cage is used, it should be connected to this ground connector. Some experiments concerning optimization of the signal-to-noise ratio can readily indicate whether or not a configuration is satisfactory.

More information on noise is provided in section 4.8.

4.6 – Autolab PGSTAT204 information

This section provides specific information for the Autolab PGSTAT204.

4.6.1 – Front panel and cell cable connections

The PGSTAT204 potentiostat/galvanostat module is installed into the Autolab PGSTAT204 instrument, alongside one optional module. From the front view, the PGSTAT204 module is always installed in the leftmost module bay. There are three connectors on the front panel of the PGSTAT204 module (see Figure 4.38).

Figure 4.38 – Overview of the PGSTAT204 module (front)

The cell cable should be plugged into the lowest socket, labelled by the symbol ⏀, on the front panel of the PGSTAT204 module. The I/O socket on the front panel can be used to connect the optional I/O cable. The DIO cable, used to connect to the optional IME663 or IME303 or for TTL triggering, can be connected to the DIO connector on the front panel.

The cell cable is labelled as follows:

- Working or indicator electrode, WE (red)
- Sense electrode, S (red)
- Reference electrode, RE (blue)
- Auxiliary or counter electrode, CE (black)

An additional ground connection (for shielding purposes, e.g. a Faraday cage) is also provided with the cell cable.

In a four electrode setup, each of the cell cable connectors is used independently. In a three electrode set-up the working electrode and sense lead are both connected to the working electrode. In a two electrode set-up the counter and reference electrode lead are both connected to the same electrode (see Figure 4.39).

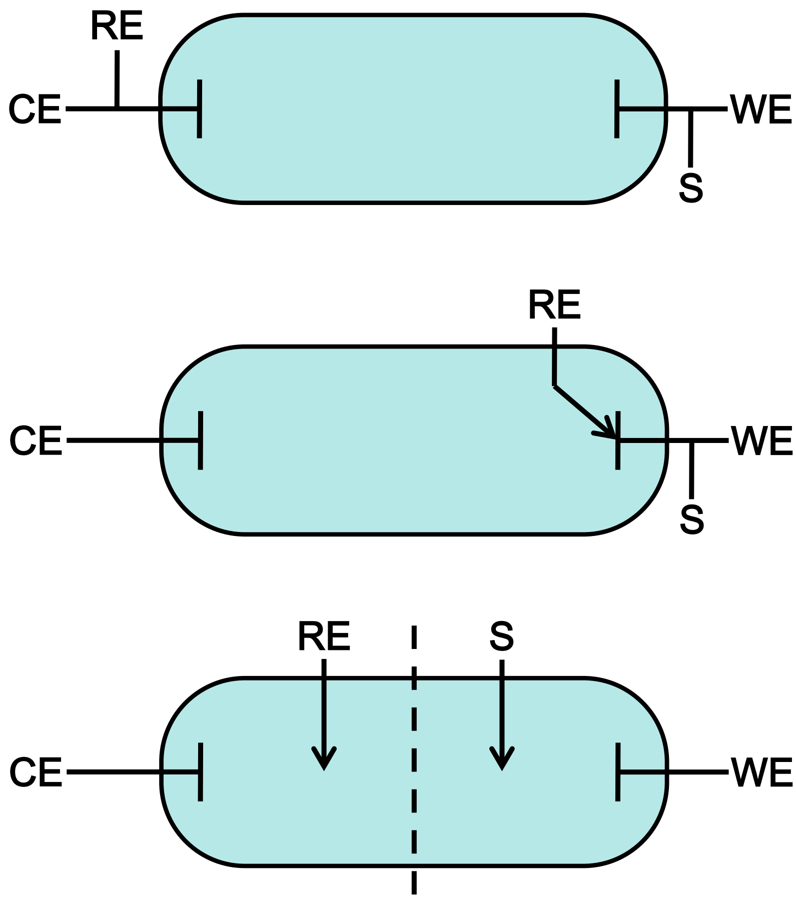


Figure 4.39 – Overview of the possible cell connections with the Autolab PGSTAT204 (from top to bottom: two electrode, three electrode and four electrode setup)

4.6.2 – Power up

The settings of the PGSTAT204 on power-up are pre-defined. The following settings are used:

- Cell: off
- Mode: Potentiostatic
- Bandwidth: High stability
- iR Compensation: off
- Current range: 1 µA

4.6.3 – Connections for analog signals

With the optional I/O cable, four additional connections are provided to the PGSTAT204 analog circuits (see Figure 4.40). All the signals are with respect to Autolab ground and indirectly to protective earth. Avoid creating ground loops as this will often degrade the performance of the PGSTAT.

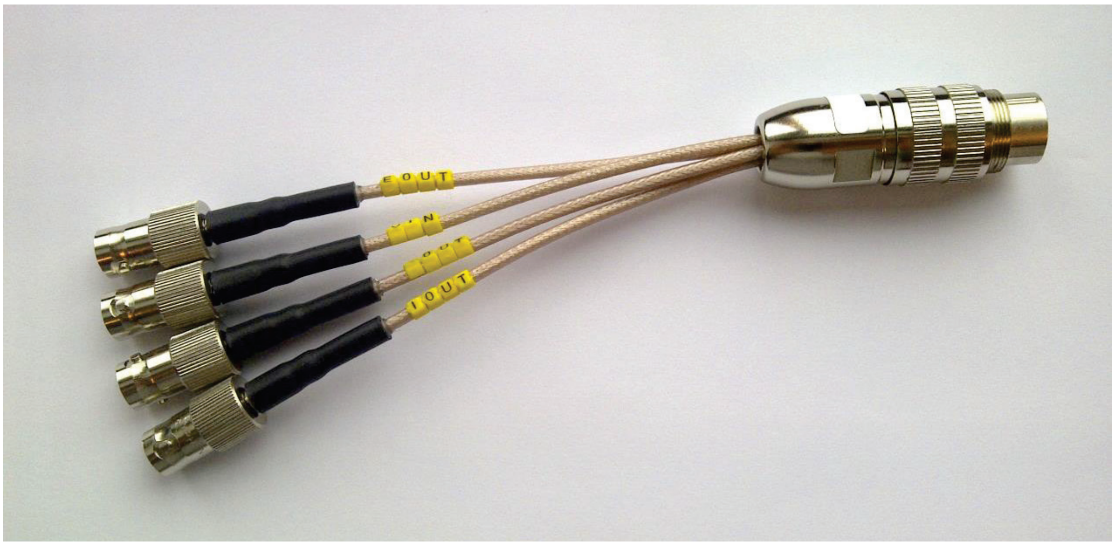


Figure 4.40 – The optional I/O cable for the PGSTAT204

The following signals are available:

E_{OUT} – This output corresponds to the differential potential of RE versus S[60]. The output voltage will vary between ±10 V. The output impedance is 1 kΩ, so a correction should be made if a load < 2 MΩ is connected. The maximum bandwidth is 300 kHz.

i_{OUT} – This signal corresponds to the inverted output of the current-to-voltage converter circuit of the PGSTAT204[61]. A 1 V signal corresponds to {-1 x the selected current range}. The output level varies between ± 10 V. The output

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[60] The E_{out} value corresponds to -WE(1).Potential.
[61] The i_{out} value corresponds to -WE(1).Current/Current range.

impedance is 50 Ω, so a correction should be made if a load < 100 kΩ is connected. The minimum load impedance is 200 Ω.

V_{OUT} – This output corresponds to the DAC output. It is controlled by software and is meant to be used to control external devices, like the rotating speed of a Rotating Disc Electrode (RDE). The output level varies between ±10 V and the output impedance is very low, < 1 Ω. The output amplifier is capable of providing 5 mA at full scale, so load impedance should be > 2 kΩ.

V_{IN} – This input corresponds to the ADC input. This input can be used for measuring a second signal. The input range is ±10 V and the input impedance is 50 Ω.

4.6.4 – High stability, High speed and Ultra high speed

The PGSTAT204 is equipped with three different bandwidth settings: High stability (HSTAB), High speed and Ultra high speed. The bandwidth can be defined using the Autolab control command (see Figure 4.41).

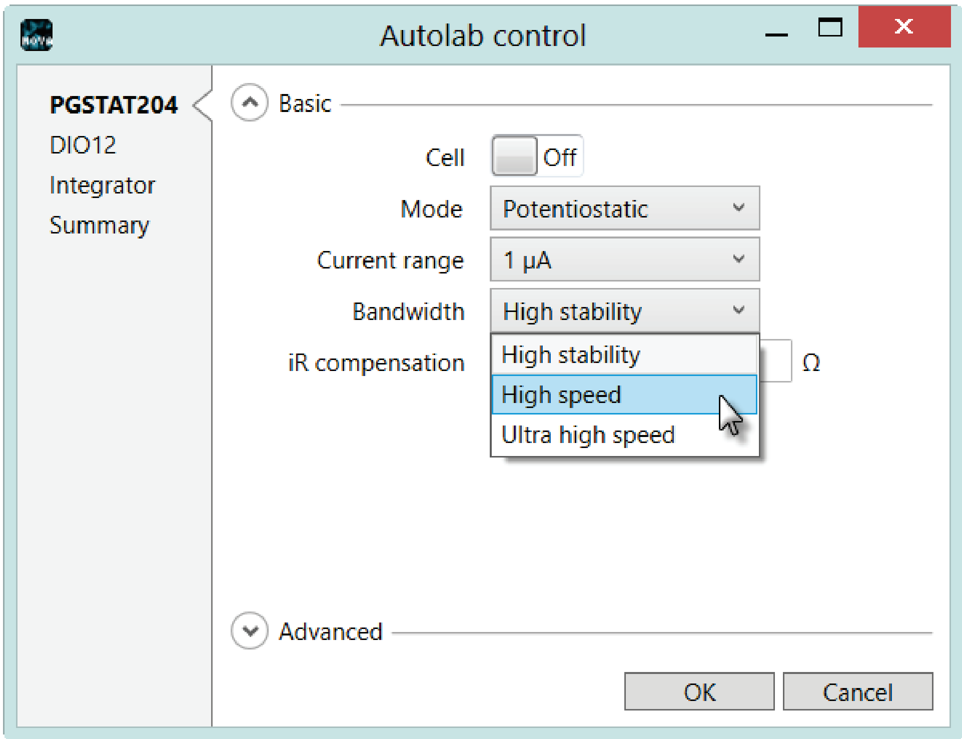


Figure 4.41 – The Autolab control window can be used to set the bandwidth of the PGSTAT204

The purpose of these different modes of operation is to provide a maximum bandwidth, maintaining stability in the PSTAT or GSTAT control loop. The normal mode of operation is High stability[62]. This gives the Control Amplifier a bandwidth of 12.5 kHz. The HSTAB indicator in the Autolab display is lit when the High stability mode is active (see Figure 4.42).

[62] Power up default setting.

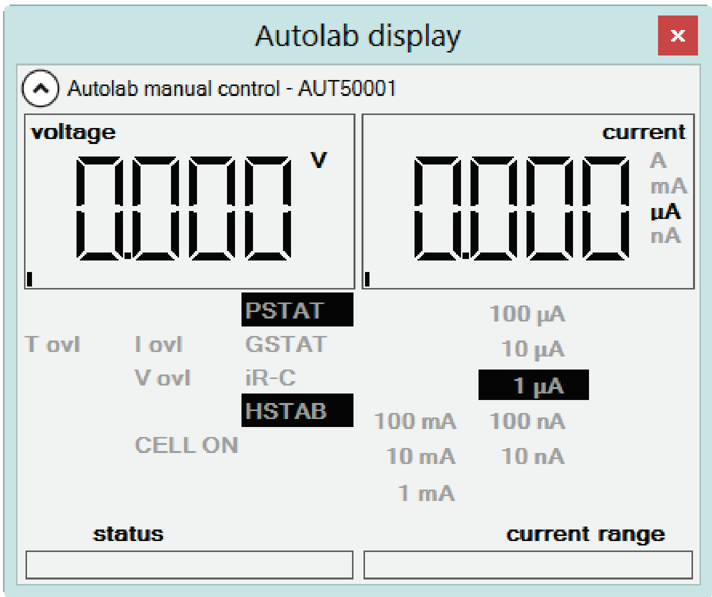


Figure 4.42 – A HSTAB indicator is provided on the Autolab display

This setting is the most appropriate for measurements at low frequencies or low scan rates. The noise in the i and E signals will be minimized. Measurements at high frequency or at high scan rates require a faster mode of operation.

When operating in High speed mode, the control amplifier will have its bandwidth extended with one decade up to 125 kHz. Some cells can show ringing or oscillation using this setting, particularly highly capacitive cells in PSTAT mode. Increasing the bandwidth also increases the noise levels for the i and E signals.

Note

It is possible to switch from High stability to High speed by clicking the HSTAB label in the Autolab display. In High speed mode, this label will be unlit on the Autolab display. Clicking the HSTAB label again switches the bandwidth back to High stability.

For applications requiring very high bandwidth, the Ultra high speed mode can be selected. In this mode, the control amplifier bandwidth is extended to 1 MHz. There is a significant oscillation risk using this setting, and the noise levels will generally show an increase relative to the High speed or High stability mode.

Warning

The higher the bandwidth, the more important it is to pay attention to adequate shielding of the cell and the electrode connectors. The use of a Faraday cage is recommended in this case.

4.6.5 – RE input impedance and stability

The electrometer RE input contains a small capacitive load. If the capacitive part of the impedance between CE and RE is comparatively large, phase shifts will occur which can lead to instability problems when working in potentiostatic mode. If the impedance between the CE and the RE cannot be changed and oscillations are observed, it is recommended to select the High stability mode to increase the system stability. In general, the use of High stability leads to a more stable control loop, compared to High speed or Ultra high speed and a significantly lower bandwidth.

To make use of the full potentiostat bandwidth (Ultra high speed mode), the impedance between CE and RE has to be lower than 35 kΩ[63]. This value is derived by testing. In galvanostat mode, this large impedance between CE and RE, will usually not lead to stability problems, because of the current feedback regulation.

4.6.6 – Galvanostat, potentiostat and iR-compensation bandwidth

For galvanostatic measurements on low current ranges, the bandwidth limiting factor becomes the current-to-voltage circuit rather than the control amplifier.

For stability reasons it is not recommended to use the High speed mode for current ranges < 10 µA. The Ultra high speed mode is also not recommended for current ranges < 1 mA.

As the current measurement circuit plays an important role in the iR compensation technique, its use is also subject to bandwidth limitations. A general indication of the maximum available bandwidth for GSTAT and for iR compensation can be found in Table 4.8:

Mode	GSTAT	iR/C - PSTAT
100 mA – 100 µA	> 1 MHz	> 1 MHz
10 µA	10 kHz	50 kHz
1 µA	10 kHz	50 kHz
100 nA	500 Hz	500 Hz
10 nA	500 Hz	500 Hz

Table 4.8 – Bandwidth overview for the PGSTAT204

At the same time, the iR-compensation bandwidth limits indicate up to which frequency current measurements can be made in potentiostatic mode (either with or without iR compensation).

4.6.7 – Galvanostatic operation and current range linearity

For galvanostatic experiments, automatic current ranging is not possible. The measurements are performed in a fixed current range. Each current range on the

[63] Empirical value.

instrument is characterized by a specific linearity limit and this specification determines the maximum current that can be applied in galvanostatic mode.

The linearity limitation also applies on measurements performed in potentiostatic mode in a fixed current range.

Table 4.9 provides an overview of the current range linearity for the PGSTAT204.

Current range	Linearity
100 mA	4
10 mA	7
1 mA	7
10 – 1 mA	7
100 – 1 µA	7
100 – 10 nA	7

Table 4.9 – Linearity limit for the PGSTAT204

For example, in the 1 mA current range, the maximum current that can be applied, galvanostatically, using the PGSTAT204, is 7 mA. The maximum current that can be measured in the 1 mA current range is 10 mA, although currents exceeding 7 mA will be measured outside of the linearity limit of this current range.

In galvanostatic operation, the applied current values are checked during the procedure validation step. When the applied current exceeds the linearity limit for the specified current range, an error message will be shown in the procedure validation screen (see Figure 4.43).

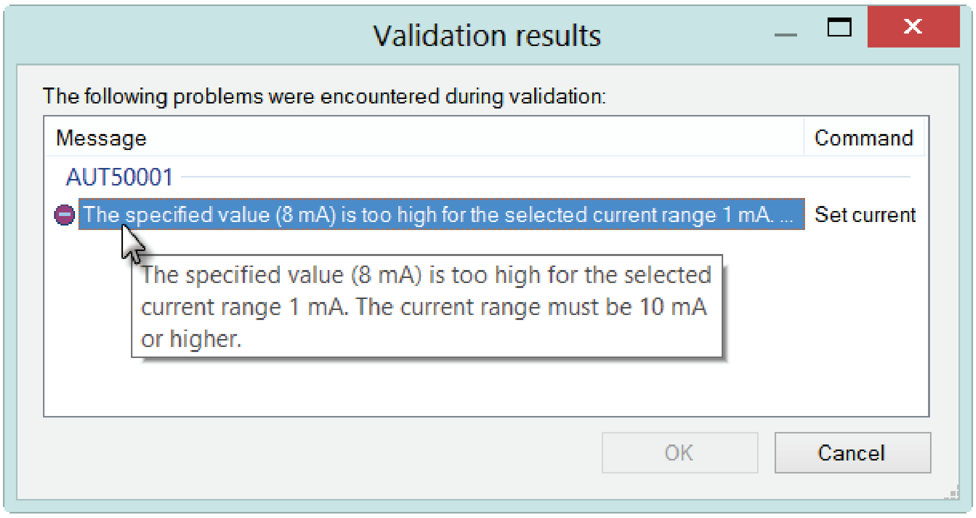


Figure 4.43 – The procedure validation step always checks the applied current values for the allowed linearity

Note

In potentiostatic mode, this check is not performed. It is possible to measure a current value in a fixed current range, even if the current value exceeds the linearity limit of the active current range. This triggers a current overload warning. When this happens during a measurement, a message will be shown in the user log, suggesting a modification of the current range (see Figure 4.44).

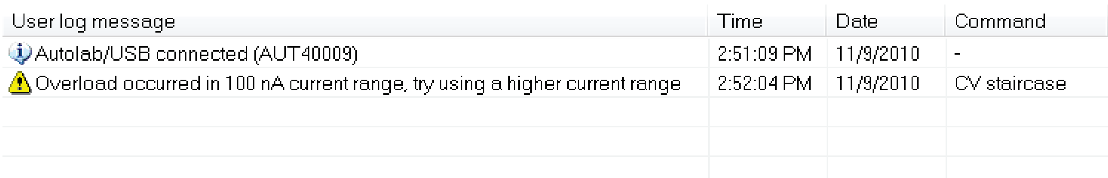


Figure 4.44 – When a current overload is detected, a suggestion is shown in the user log

4.6.8 – Maximum reference electrode voltage

The differential electrometer input contains an input protection circuitry that becomes active after crossing the ±10 V limit. This is implemented to avoid electrometer damage. The red status LED indicator on the front panel will not light up for this type of voltage overload. The measured voltage will be cutoff at an absolute value of 10.00 V.

Depending on the cell properties, galvanostatic control of the cell could lead to a potential difference between the RE and the S/WE larger than 10 V. This situation will trigger the cutoff of the measured voltage to prevent overloading the differential amplifier.

4.6.9 – Active cells

Some electrochemical cells such as batteries and fuel cells are capable of delivering power to the PGSTAT204. This is allowed only to a maximum 'cell' power, P_{MAX} of 8 W.

This means that cells showing an absolute voltage ($|V_{cell}|$) of less than 10 V between WE and CE are intrinsically safe. They may drive the PGSTAT204 output stage into current limit but will not overload the amplifier. On the other hand, cells that have an absolute voltage higher than 10 V between WE and CE may only deliver a maximum current, i_{MAX} given by:

$$i_{MAX} = \frac{P_{MAX}}{|V_{MAX}|}$$

4.6.10 – Grounded cells

The measurement circuitry of the Autolab is internally connected to protective earth (P.E.). This can be an obstacle when measurement is desired of a cell that is itself in contact with P.E. In such a case, undefined currents will flow through the loop that is formed when the electrode connections from the PGSTAT204 are linked to the cell and measurements will not be possible. Please note that not only a short circuit or a resistance can make a connection to earth, but also a capacitance is capable of providing a conductive path (for AC signals). The earth connection between the cell and P.E. should always be broken. If there is no possibility of doing this, please contact Metrohm Autolab for a custom solution, if available.

4.6.11 – Environmental conditions

The PGSTAT204 may be used at temperatures of 0 to 40 degrees Celsius. The instrument is calibrated at 25 degrees Celsius and will show minimum errors at that temperature. The ventilation holes on the bottom plate and on the rear panel may never be obstructed, nor should the instrument be placed in direct sunlight or near other sources of heat.

4.6.12 – Noise

When measuring low level currents, some precautions should be taken in order to minimize noise. The personal computer must be placed as far away as possible from the electrochemical cell and the cell cables. The cell cables should not cross other electrical cables. Other equipment with power supplies can also cause noise. For instance, the interface for mercury electrodes IME should also be placed with some care. If possible place the computer between the PGSTAT204 and other equipments. Avoid using unshielded extension cables to the electrodes. The use of a Faraday cage is also advised.

If the cell system has a ground connector, it can be connected to the analog ground connector provided with the cell cable of the PGSTAT204. If a Faraday cage is used, it should be connected to this ground connector. Some experiments concerning optimization of the signal-to-noise ratio can readily indicate whether or not a configuration is satisfactory.

More information on noise is provided in Section 4.8.

4.6.13 – Temperature overload

The PGSTAT204 is fitted with a temperature overload protection circuit. When the instrument reaches the maximum operating temperature, the protection circuit will trigger and the cell will be disconnected. The instrument will then enter in predefined safe mode and it will no longer be possible to switch the cell on. To reset the instrument, the device must be switched off, allowed to cool and then switched on again.

When the temperature overload circuit is triggered, the status LED on the front panel of the PGSTAT204 will be lit red and the corresponding indicator in the Autolab display will also be lit (see Figure 4.45).

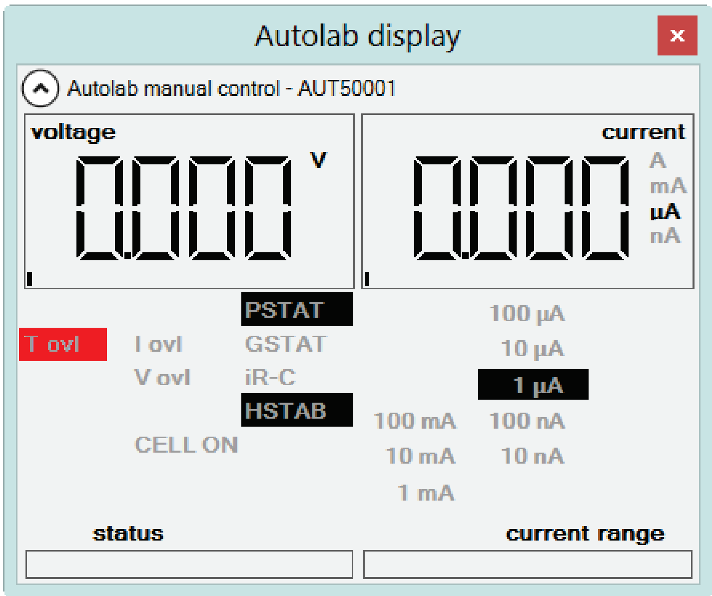


Figure 4.45 – The T ovl indicator is let when the temperature overload circuit is triggered

4.7 – µAutolab information

This section provides specific information for the µAutolab[64].

4.7.1 – Front panel and cell cable connection

There is a single connector on the front panel of the µAutolab, used to connect the cell cables (see Figure 4.46).

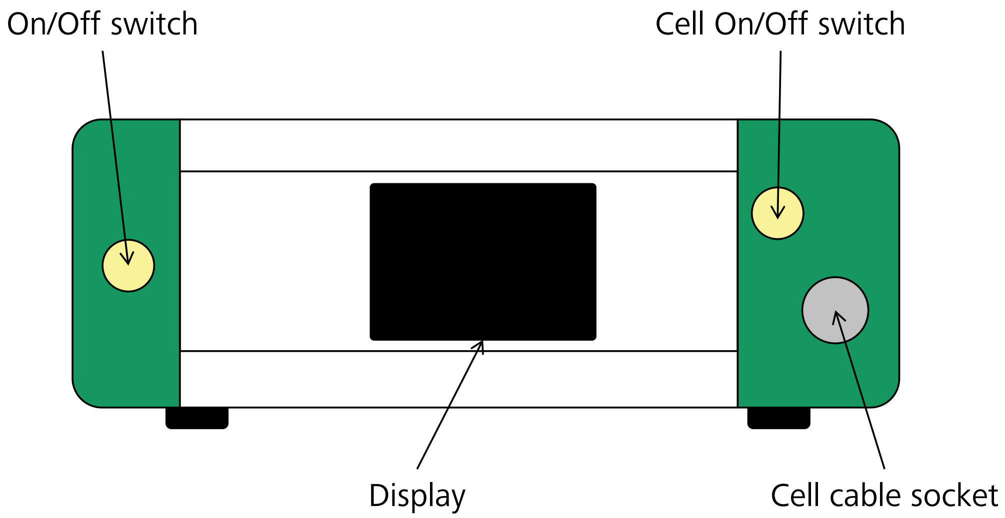


Figure 4.46 – Overview of the µAutolab

The cell cables are labelled as follows:

- Working or indicator electrode, WE (red)
- Reference electrode, RE (blue)
- Auxiliary or counter electrode, CE (black)

In a two electrode set-up the counter and reference electrode lead are both connected to the same electrode (see Figure 4.47).

[64] The µAutolab type I is not supported in NOVA.

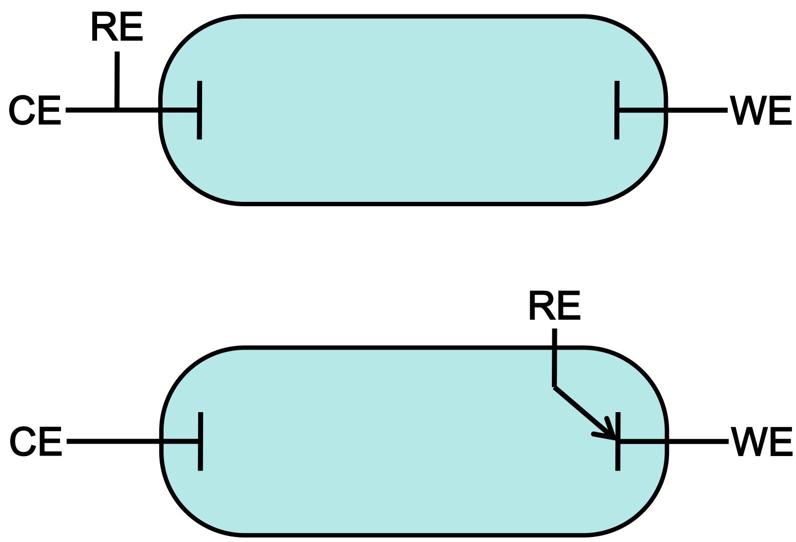


Figure 4.47 – Overview of the possible cell connections with the µAutolab (two electrode, top and three electrode setup, bottom)

4.7.2 – Power up

The settings of the µAutolab on power-up are pre-defined. The following settings are used:

- Cell: off
- Mode: Potentiostatic
- Bandwidth: High stability
- Current range: 1 µA

4.7.3 – Connections for analog signals

On the rear panel, there are four BNC connectors. All signals are with respect to µAutolab ground and indirectly to protective earth. Avoid creating ground loops as this will often degrade the performance of the instrument. From top to bottom, the following signals are available:

i_{OUT} – This signal corresponds to the output of the current-to-voltage converter circuit of the µAutolab. A 1 V signal corresponds to {1 x the selected current range}. The output level varies between ± 10 V. The output impedance is 50 Ω, so a correction should be made if a load < 100 kΩ is connected. The minimum load impedance is 200 Ω.

E_{OUT} – This output corresponds to the differential potential of RE versus S[65]. The output voltage will vary between ±10 V. The output impedance is 50 Ω, so a correction should be made if a load < 100 kΩ is connected. The minimum load impedance is 200 Ω.

[65] The E_{out} value corresponds to -WE(1).Potential.

V_{OUT} – This output corresponds to the DAC164 output. It is controlled by software and is meant to be used to control external devices, like the rotating speed of a Rotating Disc Electrode (RDE). The output level varies between ±10 V and the output impedance is very low, < 1 Ω. The output amplifier is capable of providing 5 mA at full scale, so load impedance should be > 2 kΩ.

V_{IN} – This input corresponds to the ADC164 input. This input can be used for measuring a second signal. The input range is ±10 V and the input impedance is 50 Ω.

4.7.4 – High stability and High speed

The μAutolab is equipped with two different bandwidth settings: High stability (HSTAB) and High speed. The bandwidth can be defined using the Autolab control command (see Figure 4.48).

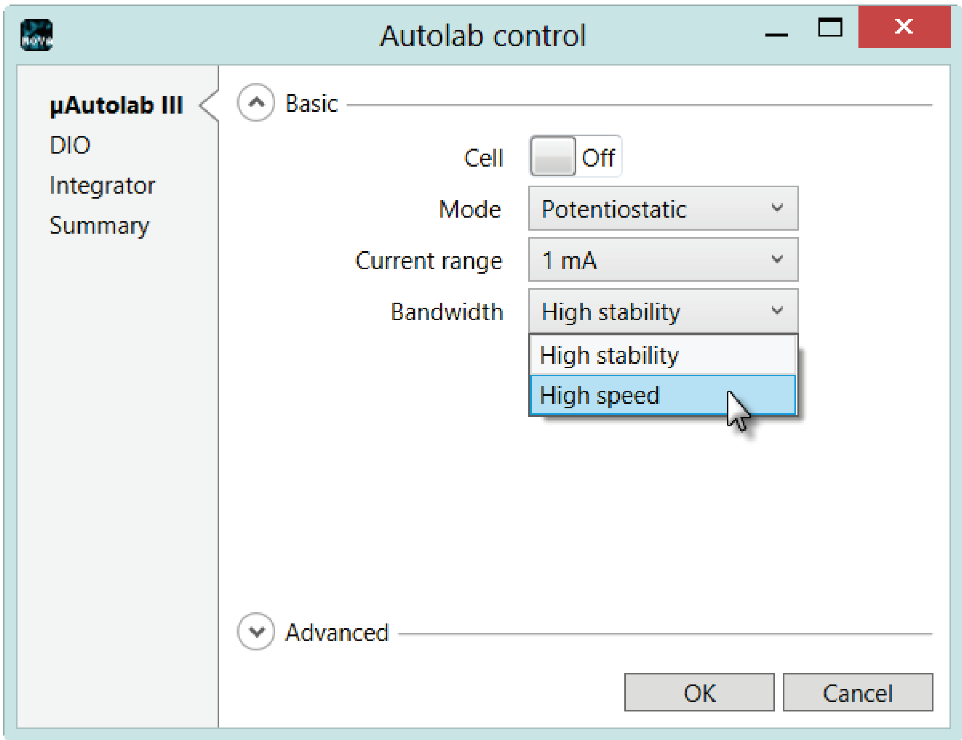


Figure 4.48 – The Autolab control window can be used to set the bandwidth of the μAutolab

The purpose of these different modes of operation is to provide a maximum bandwidth, maintaining stability in the PSTAT or GSTAT control loop. The normal mode of operation is High stability[66]. This gives the Control Amplifier a bandwidth of 12.5 kHz.

The High speed mode is automatically selected during impedance measurements at frequencies > 10 kHz, while the High stability mode is selected for frequencies below 10 kHz (see Figure 4.49).

[66] Power up default setting.

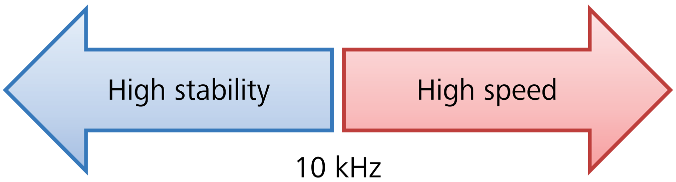


Figure 4.49 – Bandwidth limits in the µAutolab

The HSTAB indicator on the front panel of the µAutolab and in the Autolab display is lit when the High stability mode is active (see Figure 4.50).

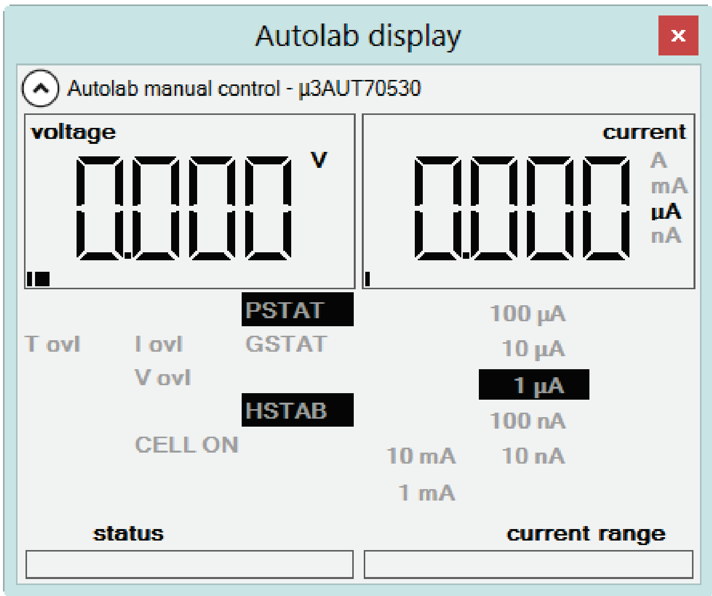


Figure 4.50 – A HSTAB indicator is provided on the Autolab display

This setting is the most appropriate for measurements at low frequencies or low scan rates. The noise in the i and E signals will be minimized. Measurements at high frequency or at high scan rates require a faster mode of operation.

When operating in High speed mode, the control amplifier will have its bandwidth extended to 500 kHz. Some cells can show ringing or oscillation using this setting, particularly highly capacitive cells in PSTAT mode. Increasing the bandwidth also increases the noise levels for the i and E signals. The High speed mode is automatically selected during impedance measurement at frequencies > 10 kHz.

Note

It is possible to switch from High stability to High speed by clicking the HSTAB label in the Autolab display. In High speed mode, this label will be unlit, both on the front panel of the µAutolab and on the Autolab display. Clicking the HSTAT label again switches the bandwidth back to High stability.

Warning

The higher the bandwidth, the more important it is to pay attention to adequate shielding of the cell and the electrode connectors. The use of a Faraday cage is recommended in this case.

4.7.5 – RE input impedance and stability

The electrometer RE input contains a small capacitive load. If the capacitive part of the impedance between CE and RE is comparatively large, phase shifts will occur which can lead to instability problems when working in potentiostatic mode. If the impedance between the CE and the RE cannot be changed and oscillations are observed, it is recommended to select the High stability mode to increase the system stability. In general, the use of High stability leads to a more stable control loop, compared to High speed and a significantly lower bandwidth.

4.7.6 – Galvanostat and bandwidth

For galvanostatic measurements on low current ranges, the bandwidth limiting factor becomes the current-to-voltage circuit rather than the control amplifier.

For stability reasons it is not recommended to use the High speed mode for current ranges < 10 µA.

A general indication of the maximum available bandwidth for GSTAT and PSTAT operation can be found in Table 4.10:

Mode	GSTAT	iR/C - PSTAT
10 mA – 1 mA	> 1 MHz	> 1 MHz
100 µA	500 kHz	500 kHz
10 µA	50 kHz	50 kHz
1 µA	5 kHz	5 kHz
100 nA	400 Hz	400 Hz
10 nA	20 Hz	20 Hz

Table 4.10 – Bandwidth overview for the µAutolab II and III

At the same time, the iR-compensation bandwidth limits indicate up to which frequency current measurements can be made in potentiostatic mode (either with or without iR compensation).

4.7.7 – Galvanostatic operation and current range linearity

For galvanostatic experiments, automatic current ranging is not possible. The measurements are performed in a fixed current range. Each current range on the instrument is characterized by a specific linearity limit and this specification determines the maximum current that can be applied in galvanostatic mode.

The linearity limitation also applies on measurements performed in potentiostatic mode in a fixed current range.

Table 4.11 provides an overview of the current range linearity for the µAutolab II and III.

Current range	Linearity
10 mA	5
1 mA	4
10 – 1 mA	4
100 – 1 µA	4
100 – 10 nA	4

Table 4.11 – Linearity limit for the µAutolab II and III

For example, in the 1 mA current range, the maximum current that can be applied, galvanostatically, using the µAutolab II or III is 4 mA. The maximum current that can be measured in the 1 mA current range is 10 mA, although currents exceeding 4 mA will be measured outside of the linearity limit of this current range.

In galvanostatic operation, the applied current values are checked during the procedure validation step. When the applied current exceeds the linearity limit for the specified current range, an error message will be shown in the procedure validation screen (see Figure 4.51).

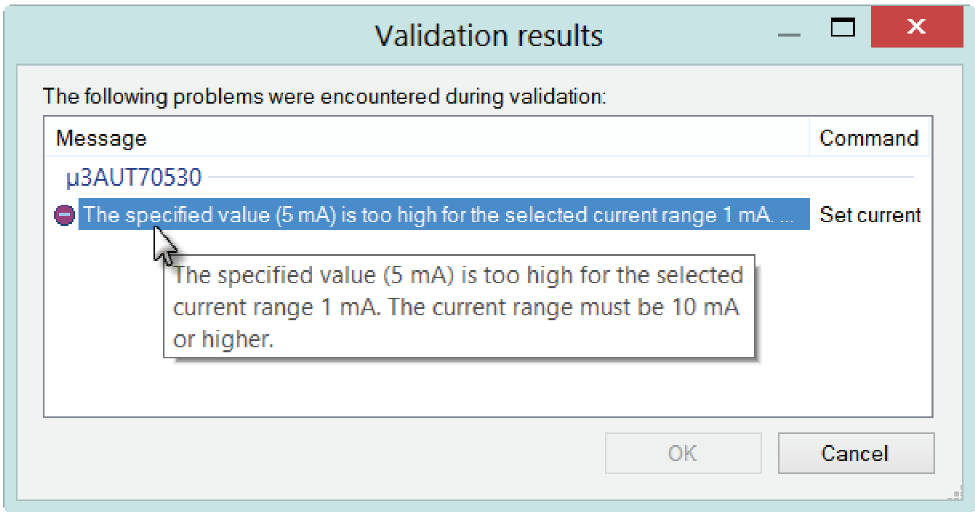
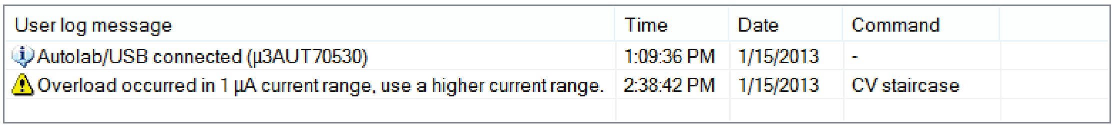


Figure 4.51 – The procedure validation step always checks the applied current values for the allowed linearity

> **Note**
>
> In potentiostatic mode, this check is not performed. It is possible to measure a current value in a fixed current range, even if the current value exceeds the linearity limit of the active current range. This triggers a current overload warning. When this happens during a measurement, a message will be shown in the user log, suggesting a modification of the current range (see Figure 4.52).



User log message	Time	Date	Command
Autolab/USB connected (µ3AUT70530)	1:09:36 PM	1/15/2013	-
Overload occurred in 1 µA current range, use a higher current range.	2:38:42 PM	1/15/2013	CV staircase

Figure 4.52 – When a current overload is detected, a suggestion is shown in the user log

> **Note**
>
> The maximum measurable current with the µAutolab II and III is 80 mA.

4.7.8 – Maximum reference electrode voltage

The electrometer RE input contains an input protection circuitry and becomes active after crossing the ±10 V limit. This is implemented to avoid electrometer damage. Please note that the V_{ovl} indicator will not light up for this type of voltage overload. The measured voltage will be cutoff at an absolute value of 10.00 V.

Depending on the cell properties, galvanostatic control of the cell could lead to a potential difference between the RE and the WE larger than 10 V. This situation

will trigger the cutoff of the measured voltage to prevent overloading the differential amplifier.

4.7.9 – Active cells

Some electrochemical cells such as batteries and fuel cells are capable of delivering power to the µAutolab. This is allowed only to a maximum 'cell' power, P_{MAX} of 0.5 W.

This means that cells which show an absolute voltage ($|V_{cell}|$) of less than 5 V between WE and CE are intrinsically safe. They may drive the PGSTAT output stage into current limit but will not overload the amplifier. On the other hand, cells that have an absolute voltage higher than 5 V between WE and CE may only deliver a maximum current, i_{MAX} given by:

$$i_{MAX} = \frac{P_{MAX}}{|V_{MAX}|}$$

4.7.10 – Grounded cells

The measurement circuitry of the µAutolab is internally connected to protective earth (P.E.). This can be an obstacle when measurement is desired of a cell that is itself in contact with P.E. In such a case, undefined currents will flow through the loop that is formed when the electrode connections from the µAutolab are linked to the cell and measurements will not be possible. Please note that not only a short circuit or a resistance can make a connection to earth, but also a capacitance is capable of providing a conductive path (for AC signals). The earth connection between the cell and P.E. should always be broken. If there is no possibility of doing this, please contact Metrohm Autolab for custom solution, if available.

4.7.11 – Environmental conditions

The µAutolab may be used at temperatures of 0 to 40 degrees Celsius. The instrument is calibrated at 25 degrees Celsius and will show minimum errors at that temperature. The ventilation hole on the rear panel may never be obstructed, nor should the instrument be placed in direct sunlight or near other sources of heat.

4.7.12 – Noise

When measuring low level currents, some precautions should be taken in order to minimize noise. The personal computer must be placed as far away as possible from the electrochemical cell and the cell cables. The cell cables should not cross other electrical cables. Other equipment with power supplies can also cause noise. For instance, the interface for mercury electrodes IME should also be placed with some care. If possible place the computer between the µAutolab and other equipments. Avoid using unshielded extension cables to the electrodes. The use of a Faraday cage is also advised.

If the cell system has a ground connector, it can be connected to the analog ground connector at the front of the µAutolab. If a Faraday cage is used, it should be connected to this ground connector. Some experiments concerning optimization of the signal-to-noise ratio can readily indicate whether or not a configuration is satisfactory. More information on noise is provided in Section 4.8.

4.8 – Noise considerations

The high sensitivity of the Autolab potentiostat/galvanostat makes it susceptible to noise pickup. In particular, the noise coming from the mains power can cause severe disturbance in the measurements.

4.8.1 – Problems with the reference electrode

If the reference electrode is not filled properly with electrolyte solution or when it has, for other reasons, a very high impedance, it will be expressed as noise. In most cases the applied potential is not the same as the measured potential. Refer to the user manual provided by the reference electrode supplier for more information on the proper care of your reference electrode.

4.8.2 – Problems with unshielded cables

It is not advisable to use unshielded electrode cables. Make the connections to the electrodes as close as possible to the electrode itself. Avoid the use of unshielded extension cables to the electrodes.

4.8.3 – Faraday cage

The use of a Faraday cage is always recommended. It protects the cell from external noise interference. Connect the cage to the green ground-connector of the cell cable or to ground connector GND at rear of the Autolab instrument.

4.8.4 – Grounding of the instrument

Not proper grounding of the Autolab and PC will decrease the signal to noise ratio. Always use a grounded power-point and grounded power-cables. Be sure to connect the Autolab and PC to the same power ground. This means they should be connected to the same outlet.

4.8.5 – Magnetic stirrer

In some cases a magnetic stirrer can cause noise problems. Try the measurements with the stirrer on and off and monitor the current. If the stirrer causes a lot of noise please try to find another way of stirring.

4.8.6 – Position of the cell, Autolab and accessories

The signal to noise ratio can often be improved by changing the positions of the cell, computer and ancillary equipment relative to the Autolab. In general, the electrochemical cell should be placed as far as possible from the computer and

other devices, without extending the cell cables with unshielded cables. If the noise level remains too high, a Faraday cage may be necessary.

4.8.7 – Measurements in a glove box

When the cell needs to be place into a glove box, it is highly recommended to use feed through that allows the Autolab cell cables to be connected to the cell inside the glove box. If necessary, the cell cables of the Autolab can be fitted with BNC connectors rather than 4 mm banana connectors. This allows using BNC feedthroughs. Contact your Autolab distributor for more information about this modification.

Note

The shielding of the RE and S cable on the PGSTAT and of the RE cable on the μAutolab is driven (or guarded). Use isolated cable feedthroughs for these cables in order to extend the driven shield inside the glove box. The shield of these cables must not be connected to the ground of the glove box.

4.9 – Cleaning and inspection

It is recommended to clean the Autolab cabinet and the accessories on a regular basis. This can be done with a damp cloth, optionally using a mild detergent. Never use an excessive amount of water; it may never enter into the instrument. As a precaution, disconnect Autolab from the mains when cleaning it. Also perform an inspection of the instrument and all of the connecting cables. If you find any cables with damaged insulation or other irregularities, stop using the instrument until it has been repaired.

Damaged equipment or damaged cables may be hazardous!

Warranty and conformity

This chapter provides information about warranty, safety, specifications and conformity.

5 – Warranty and conformity

5.1 – Safety practices

Please read the safety practices carefully before starting to use the AUTOLAB instrument

This section describes the AUTOLAB instrument. The section deals with appearance and use of the instrument and contains necessary information regarding operation and installation. Not following these instructions when using AUTOLAB may cause unsafe operation.

General

The following safety practices are intended to ensure safe operation of the equipment. Not following these instructions when using Autolab may cause unsafe operation. Metrohm Autolab is **not liable** for any damage caused by not complying with the following instructions.

Electrical Hazards

1. There are no user-serviceable parts inside; servicing should only be done by qualified personnel.
2. Removal of panels exposes to potentially dangerous voltages. Always disconnect the instrument from all power sources before removing protective panels.
3. Replace blown fuses only with size and rating stipulated on or near the fuse panel holder and in the manual.
4. Replace or repair faulty or frayed insulation on power cords and control cables.
5. Replace control cables only with original spare parts.
6. When replacing power cord, use only approved type and conform local regulations.
7. Be sure power cords are plugged into the correct voltage source and always use a wall outlet with protective earth.
8. Check all connected equipment for proper grounding. Do not move the instrument with power cords connected.

General Precautions

1. Do not place the instrument on an unstable surface.
2. Do not expose the instrument to damp or wet conditions.

3. To prevent overheating, care should be taken not to cover any of the instrument ventilation holes and not to place the instrument close to a heating source.
4. Full EMC compliance can only be achieved when the electrochemical cell is placed inside a Faraday cage.

5.2 – General specifications

Power Supply (V)	Booster20A, μAutolab type III, μAutolab type III/FRA2, PGSTAT101, PGSTAT204	100-240V ± 10% (auto select)
	BSTR10A	100-240V ± 10% selectable in 2 ranges 100:92..132V 240:198..264V
	IME303, IME663	100-240V ± 10% selectable in 4 ranges 100: 90..121V 120: 104..139V 220: 198..242V 240: 207..264V
	PGSTAT302N, PGSTAT302F, PGSTAT128N, PGSTAT100N	100-240V ± 10% selectable in 4 ranges 100: 90..121V 120: 104..139V 230: 198..242V 240: 207..264V
	Multi Autolab	100-240V ± 10% (auto select)
Power-Line frequency	47-63 Hz	
Power consumption (VA max.)	μAutolab type III, μAutolab type III/FRA2	144
	PGSTAT101	40
	PGSTAT204	75
	Multi Autolab (12 M101)	200
	PGSTAT302N, PGSTAT302F	300
	PGSTAT128N	180
	PGSTAT100N	247
	Booster20A	950
	BSTR10A	650
	IME303, IME663	50

Fuse (A, slow-slow)	μAutolab type III	1.6				
	μAutolab type III/FRA2	1.6				
	PGSTAT101	2				
	PGSTAT204	3.5				
	Booster20A	8				
		100V	**120V**	**220V**	**230V**	**240V**
	PGSTAT302N, PGSTAT302F, PGSTAT128N	3.15	3.15		1.6	1.6
	PGSTAT100N	3.15	3.15		1.25	1.25
	BSTR10A		4			4
	IME303, IME663	630 m	630 m	315 m		315 m
Fuse (A, fast)	Multi Autolab (refer to backplane)	5 or 8				

Operating Environment	0 °C to 40 °C ambient temperature without derating, 80% relative humidity
Storage Environment	-10 °C to + 60 °C ambient temperature

Dimensions (W x H x D)	µAutolab type III, µAutolab type III/FRA2	27 x 27 x 9 cm^3
	PGSTAT101	9 x 21 x 15 cm^3
	PGSTAT204	15 x 26 x 20 cm^3
	PGSTAT302N, PGSTAT302F, PGSTAT128N, PGSTAT100N	52 x 42 x 17 cm^3
	Booster20	52 x 49 x 20 cm^3
	BSTR10A	36 x 47 x 15 cm^3
	IME303, IME663	20 x 24 x 8 cm^3
	Multi Autolab	52 x 42 x 17 cm^3
Weight (kg)	µAutolab type III	3.6
	µAutolab type III/FRA2	4.2
	PGSTAT101	2.1
	PGSTAT204	4.1
	PGSTAT302N, PGSTAT302F	18
	PGSTAT128N	16
	PGSTAT100N	21
	Booster20A	25
	BSTR10A	9
	IME303, IME663	2.7
	Multi Autolab	12.8

Safety designed to	EN61010-1
EMC compliance	EN61326-1, EN61326-1 *Note:* full EMC compliance with all cell types can only be achieved with cell placed in a faraday cage
Warm-up time	30 minutes
Remote interface	USB
Pollution degree	2
Installation category	II

5.3 – Warranty

The warranty on Autolab products is limited to defects that are traceable to material, construction or manufacturing error, which occur within 36 months from the day of delivery (12 months for instruments delivered before January 1st, 2012). In this case, the defects will be rectified by Metrohm Autolab free of charge. Transport costs are to be paid by the customer.

Glass breakage in the case of electrodes, cells or other parts is not covered by the warranty. Consumables (electrodes, QCM crystals, etc.) are not covered by the warranty.

If damage of the packaging is evident on receipt of the goods or if the goods show signs of transport damage after unpacking, the carrier must be informed

immediately and a written damage report is demanded. Lack of an official damage report releases Metrohm Autolab from any liability to pay compensation.

If any instruments or parts have to be returned, the original packaging should be used. This applies to all instruments, electrodes, cells and other parts. If the original packaging is not available it can be ordered at Metrohm Autolab or at your local distributor. For damage that arises as a result of non-compliance with these instructions, no warranty responsibility whatsoever will be accepted by Metrohm Autolab.

Do not modify the cell cable or the differential amplifier cable connectors. These cables are designed for the best possible operation. Modifications of these connections, i.e. with other connectors, will lead to the loss of any warranty.

5.4 – EU Declaration of conformity

Declaration of Conformity

This is to certify the conformity to the standard specifications for electrical appliances and accessories, as well as to the standard specifications for security and to system validation issued by the manufacturing company.

Name of commodity

AUTOLAB

Metrohm Autolab B.V.
Utrecht, The Netherlands
info@metrohm-autolab.com
www.metrohm-autolab.com

Description Instrument for electrochemical research and voltammetric analysis.

This instrument is designed and tested according to the standards:

Electromagnetic compatibility: Emission
EN 61326-1 (1997) + A1 (1998) + A2 (2001) + A3 (2003)
EN 61000-3-2 (2006)
EN 61000-3-3 (1995) + A1 (2001) + A2 (2005)

Electromagnetic compatibility: Immunity
EN 61326-1 (1997) + A1 (1998) + A2 (2001) + A3 (2003)

Safety specifications
EN 61010-1

CE

The instrument is in conformity with EU directives 89/336/EEC and 73/23/EEC and fulfils the following specifications:

EN 61326	Electrical equipment for measurement, control and laboratory use – EMC requirements.
EN 61010-1	Safety requirements for electrical equipment for measurement, control and laboratory use.

Metrohm Autolab B.V. is holder of the TŰV-certificate of the quality system ISO 9001:2008 for quality assurance in development, production, sales and service of instruments and accessories for electrochemistry and biochemistry (registration number 7528/2.2).

Utrecht, 1 October 2009

J.J.M. Coenen
QC Manager

A. Idzerda
Head of Production

Metrohm Autolab B.V. at Utrecht will not accept any liability for damages caused directly or indirectly by connecting this instrument to devices which do not meet relevant safety standards. AUTOLAB was developed as a laboratory research instrument. Metrohm Autolab B.V. cannot under any circumstance be held responsible for the outcome or interpretations of data measured with AUTOLAB.

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03/2013

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